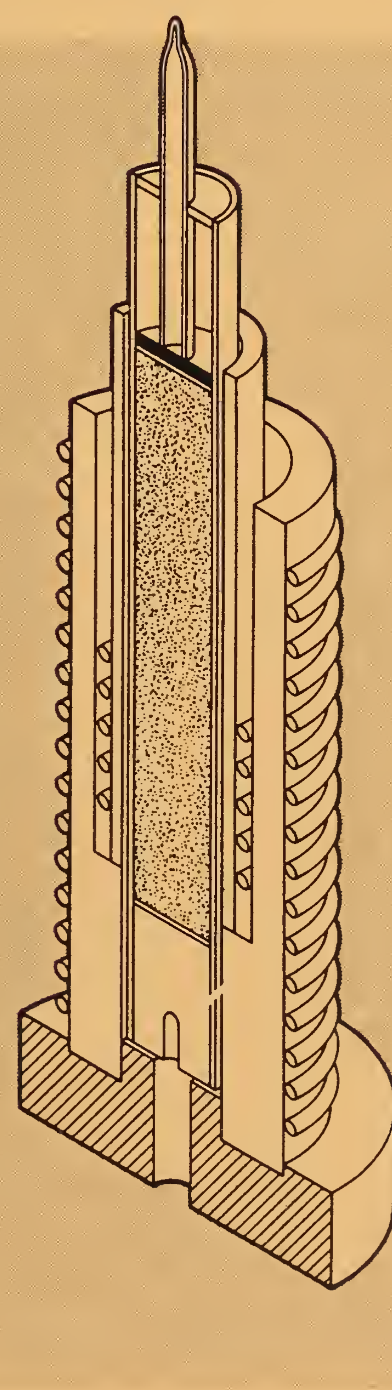
IMSE

Institute for Materials Science and Engineering

METALLURGY



NAS-NRC
Assessment Panel
February 2–3, 1989

NISTIR 88-3843
U.S. Department of Commerce
National Institute of Standards
and Technology

Technical Activities 1988

Eddy Current Sensor used for measuring the density of metal samples in real-time during hot-isostatic-pressing (HIP).

Institute for Materials Science and Engineering

METALLURGY

E.N. Pugh, Chief
J.H. Smith, Deputy

NAS-NRC
Assessment Panel
February 2–3, 1989

NISTIR 88-3843
U.S. Department of Commerce
National Institute of Standards and Technology

Technical Activities 1988

METALLURGY DIVISION

CHIEF

E. Neville Pugh
Phone: (301) 975-5960

DEPUTY CHIEF

John H. Smith
Phone: (301) 975-5961

SPECIAL ASSISTANT

Steve C. Hardy
Phone: (301) 975-6159

GROUP LEADERS

Advanced Sensing
Haydn N. G. Wadley
Phone: (301) 975-6140

Corrosion
Dale E. Hall
Phone: (301) 975-6022

Electrodeposition
David S. Lashmore
Phone: (301) 975-6405

High Temperature Materials Chemistry
John W. Hastie
Phone: (301) 975-5754

Magnetic Materials
Lawrence H. Bennett
Phone: (301) 975-5966

Mechanical Properties of Metals
John H. Smith
Phone: (301) 975-5961

Metallurgical Processing
John R. Manning
Phone: (301) 975-6157

ABSTRACT

This report summarizes the FY 1988 activities of the Metallurgy Division of the National Institute of Standards and Technology (NIST). These activities center upon the structure-processing-properties relations of metals and alloys and on methods of measurement; and also include the generation and evaluation of critical materials data. Efforts comprise studies of metals processing and process sensors; advanced materials, including metal matrix composites, intermetallic alloys and superconductors; corrosion and electrodeposition; mechanical properties; magnetic materials; and high temperature reactions.

The work described also includes four cooperative programs with professional societies and industry: the Alloy Phase Diagram Program (with ASM INTERNATIONAL); the Corrosion Data Program (the National Association of Corrosion Engineers); the Steel Sensor Program (the American Iron and Steel Institute); and the Temperature Sensor Program (the Aluminum Association).

The scientific publications, committee participation, and other professional interactions of the 79 full-time and part-time permanent members of the Metallurgy Division and its 48 guest researchers are identified.

TABLE OF CONTENTS

OVERVIEW

METALLURGY DIVISION (450)

E. Neville Pugh, Chief
John H. Smith, Deputy Chief
June Toms, Secretary

The basic programs in the Metallurgy Division fall into the traditional NIST areas of measurement science, materials characterization, and data and SRMs, but many have undergone radical changes over the past few years. In response to a clear mandate to assist in increasing the nation's industrial competitiveness, emphasis in measurement science continues to move towards processing, specifically to process sensors and, in some instances, to process modeling and automated process control. The focus on processing has also led to a more direct interaction with industry, as evidenced by the consortium for powder atomization and other joint programs. In materials characterization, the thrust is towards new, advanced materials, including metal matrix composites, intermetallic alloys, and nanocrystalline magnetic materials, as well as high T_c superconductors. The Corrosion Data Program (with NACE) has replaced the successful Alloy Phase Diagram Program (with ASM International) as our largest data activity. It will be seen in the following summary that there is considerable cross cutting between the main program areas, and that many programs involve strong interactions between the seven groups which constitute the Division.

The major programs are conveniently grouped into three categories:

Metals Processing and Sensors

Powder processing represents a major current thrust, with programs involving both powder atomization and consolidation. The former centers on the Metallurgical Processing Group's high pressure inert gas atomizer, and involves a cooperative effort with industry in the form of a consortium and with other centers in NIST. This program is directed towards the measurement and real-time control of particle size, such control being important for efficient consolidation and to obtain desired properties, particularly in RSP applications. In the first year of the consortium, approximately twenty 10Kg batches of Type 304 steel were atomized, both to determine the effects of parameters such as die pressure, the ratio of the gas and metal flow rates, and metal superheat, and to study in-situ particle size measurement techniques. Particle size and distribution was also measured off-line by sieving. At present, a laser diffraction technique shows promise as an in-situ sensor. Modeling of the atomization process requires detailed information on the way in which the molten stream breaks into fine droplets, and direct studies of the plume have been initiated using high speed photograhy and laser holography. These studies are being carried out cooperatively with fluid mechanics experts from the Center for Chemical Engineering.

Powder consolidation studies focus on hot isostatic pressing (HIP). In a program jointly sponsored by DARPA and carried out in the Advanced Sensing

and Metallurgical Processing Groups, sensors are being developed to monitor the consolidation process in real time. These studies are aimed at Ti-Al intermetallics but preliminary studies have also been carried out on copper. Considerable progress has been made with an eddy current sensor to measure shrinkage as densification proceeds; this technique permits dimensional changes of a few microns to be measured. An ultrasonic sensor to monitor microstructural changes is in an earlier stage of development. A theoretical model of the HIP process has been developed by Ashby, University of Cambridge, who serves as a consultant to the project. The experimental data have revealed several ways in which the model should be modified to give a more complete description of the HIP process. In the final stage of the program, an intelligent control system, currently being developed by the BDM Corporation, will compare the sensor data to the predictions of the upgraded theoretical model, and recommend changes in the process schedule. By this means it should be possible to obtain an end product with the desired characteristics even in the presence of variations in the starting material.

Other sensors are being studied in the Advanced Sensing Group. The eddy current technique mentioned in the HIP project is also being used in a collaborative program with the Aluminum Association to measure the internal temperature of aluminum during extrusion. A plant demonstration in 1987 demonstrated the practicality of this non-contact method for cylindrical and square-section products, and it is now being extended to more complex shapes where through-transmission must be used. Further demonstrations are planned for later in 1988, one at a plant and one at NIST. Work has also continued on the ultrasonic time of flight system being developed with the American Iron and Steel Institute. Earlier laboratory tests demonstrated the feasibility of the method for measuring the internal temperature of solid steel, and it is now being evaluated in field tests. In the laboratory, the approach is now being extended to determine the position of the liquid-solid interface in partially solidified material, knowledge necessary for processes such as continuous casting and skull melting. To date this work has been carried out largely on aluminum because of its relatively low melting point, and the results have been encouraging. Studies on Rene 95 are now in progress.

The High Temperature Materials Chemistry Group is continuing its development of thermochemical computer models for liquid iron and steel processing. Such models are particularly timely in view of recent renewed interest by industry in developing radically new direct reduction steel making processes. The modeling research is supported by solution thermochemical data obtained using an unique high pressure, high temperature mass spectrometer facility which allows measurements to be made at temperatures, pressures and compositions representative of steel processing conditions.

Characterization of Advanced Materials

Metal matrix composites (MMC), represent an important emerging technology in which we have mounted a significant effort, cutting across several groups. The bulk of the work so far has been carried out on SiC fibers in matrices of aluminum or its alloys, and has focussed on the fiber-matrix interface, which controls the performance of the composites. In the Metallurgical Processing Group, TEM studies are being conducted to determine the interface structures

corresponding to conditions simulating molten metal infiltration, and these are correlated with phase diagram calculations of equilibrium and non-equilibrium phase formation; the results have shown, for example, that the brittle intermetallics Al_4C_3 and Al_4SiC_4 can form under some conditions, and that the morphologies of the Al-SiC and Al_4C_3-SiC interfaces are strongly influenced by the SiC grain structure. As part of the studies, a technique has been perfected to grow, from the melt, Al monocrystals containing a single SiC fiber. These samples have been used by the Advanced Sensing Group to study the mechanical behavior of the interface for different thermal histories, using the acoustic emission technique to monitor deformation, and to examine the perfection of the interface with an ultrasonic "leaky wave" method.

The Electrodeposition Group is continuing its novel approach to composites, using electrodeposition to coat the fibers with an Al alloy, possibly with an intermediate layer to optimize the interfacial structure or as a barrier for improved performance at elevated temperatures. The coated fibers may then be consolidated by conventional infiltration techniques, or the matrix may be built up by electroforming followed by a hot pressing operation. A cell for alloy deposition on moving fibers has been completed, and the process has been successfully modeled. Over the past year, effort has focussed on depositing and characterizing Al-Mn matrixes, the manganese enhancing the nucleation process, and Al-Ti matrices for intermetallic composites. Results to date are promising for both systems. In related cooperative work with the American Cyanamid Company, Co-W layers have been successfully plated onto graphite fibers to produce effective barrier layers.

Intermetallic alloys represent another major activity in this program area. It was seen that HIP studies and alloy electrodeposition are being carried out on Ti-Al alloys, which are potentially important for high temperature structural applications. A major DARPA supported program on ternary alloys based on this system is in progress in the Metallurgical Processing Group. This research is directed towards the examination of processing paths using precursor metastable phases produced by rapid liquid and solid state quenching, an approach which offers possibilities of microstructural manipulation beyond those available by conventional processing. For example, rapid solidification of Ni_2TiAl (normally the $L2_1$ structure) has caused the formation of an ordered BCC (B2) phase, and subsequent heat treatment causes the formation of $L2_1$ cuboids in a B2 matrix. A general theory has been constructed to predict the formation of such unusual microstructures. Solid state quenching experiments are being conducted in the Ti-Al-Nb system. Examination of phase equilibria and transformations has led to the delineation of a large ternary field of ordered BCC which is thought to be important in establishing the properties of the alloy system. Decomposition of the quenched B2 phase is currently being investigated.

Nickel aluminides are being studied in the Corrosion Group. In this program, materials supplied by Oak Ridge National Laboratories are being characterized for resistance to aqueous corrosion and environment induced embrittlement. Work to date has established that the alloys display good resistance to general and pitting corrosion in neutral and alkaline solutions, but undergo severe hydrogen embrittlement in acid conditions. Further work is in

progress to determine if localized attack can produce acidification in neutral solutions which might lead to hydrogen absorption.

A new activity has been undertaken by the High Temperature Materials Chemistry Group on the oxidation mechanisms for refractory intermetallics and composites. The program is supported by DARPA, and an industrial advisory panel provides guidance on the research directions. Initial studies involve determination of the basic thermochemistry of Nb-Al-O, a system of central importance to the development of refractory structures for hypersonic vehicles.

Advanced magnetic and superconducting materials have been characterized in the Magnetic Materials Group's newly modernized magnetic materials laboratory. High T_c superconductors based on either rare earths, bismuth or thallium, have been measured as bulk materials and as laser-ablated thin films. Magnetic hysteresis, magnetic viscosity, flux creep, superconducting onset temperatures, magnetic critical currents, and in iron-substituted samples, magnetic hyperfine fields have all been investigated. The thin films were measured using a novel magnetically-modulated-microwave-absorption (MAMMA) technique. The High Temperature Materials Chemistry Group has started investigations on the detailed character of the "plume" in the laser-ablation process. Magnetic measurements on Ni/Cu compositionally-modulated thin films produced by the Electrodeposition Group with their improved electroplating procedure exhibited (1) high magnetizations and (2) smaller temperature dependencies of the magnetization than any reported previously by any technique. These results demonstrate that electrodepositing nanometer thick layers of the highest quality is feasible. Thin films of a magnetic and a nonmagnetic constituent with a nanometer-sized granular morphology have been produced both by sputtering and by a sol-gel method. These nanocomposites were characterized by Mossbauer spectroscopy, x-ray diffraction and, of special importance, field-emission scanning-electron microscopy (FESEM).

<u>Data and SRM Programs</u>

The Alloy Phase Diagram Program on binary alloys, conducted cooperatively with ASM International, is drawing to a successful conclusion. Development and loading of the interactive database containing numerical and graphical data for nearly 1,600 binary systems was completed in 1988 and the database transferred to ASM for dissemination. In addition to providing the Editor of the Bulletin of Alloy Phase Diagrams, the Division's remaining activity in the binary program is the evaluation of the iron binaries. The main thrust of our program is now in ternary diagrams, both in evaluation of certain critical systems and in developing a ternary graphics package, and in providing support for other in-house programs such as those described above on MMC and intermetallic alloys.

The Corrosion Data Center, formed in cooperation with NACE in response to a critical need for a centralized, computerized database, continued to expand in 1988. CORSUR, a program containing a NACE survey of data for metals and non-metals, was completed and is being distributed by NACE. A comprehensive database designed to accommodate data from a variety of sources has been developed, and data from two key industrial sources are currently being

entered for subsequent evaluation. In addition, expert systems are under development in two areas of materials selection, one for the handling and storage of concentrated sulfuric acid (with MTI) and the other for sub-surface pumps for oil and gas production (with a Guest Scientist from the New Zealand DSIR).

Another major data program is being conducted for the Nuclear Regulatory Commission in their high level waste management program. Specifically, the Corrosion Group assists NRC in evaluating DOE's activities in waste disposal, particularly in areas relating to the localized corrosion of the waste package container. Technical reviews of DOE's technical reports and other papers are being entered into a computerized database. An experimental program is also being conducted, primarily to resolve issues arising from the reviewing process.

The Division continues to maintain a strong program in Standard Reference Materials. In the Electrodeposition Group, production of coating thickness standards increased 10% in the past year, and research continued on the development of a lead-tin standard for the electronics industry. The Mechanical Properties of Metals Group continued a project with ASTM to develop test blocks for use in the calibration of Rockwell hardness machines. An important finding in 1988 was that variations in indenter geometry contribute significantly to the variability of certain hardness measurements previously attributed to the test blocks.

METALLURGY DIVISION

National Institute of Standards and Technology
Institute for Materials Science
and Engineering

Administrative Office
M. L. Slusser
S. L. Neal

Division Chief: E. N. Pugh
Deputy Chief: J. H. Smith
Special Assistant: S. C. Hardy
Secretary: L. J. Toms
Clerk Typist: N. E. Wilkerson

Institute Scientist
J. W. Cahn

ELECTRODEPOSITION**

Ldr: D. S. Lashmore
Secy: S. L. Becker

Beauchamp, C. R.
Brown, H. G.
Brown, H. J.
Claggett, S. A.
Johnson, C. E.
Kelley, D. R.
Mullen, J. L.
Oberle, R. R.
Sharpless, P. N.
Stafford, G. R.

Students
Boulia, D. J.
Stewart, G. D.
Nyhus, P. A.

Guest Scientists
Ogburn, F.
Rosset, E.

Research Associate
Wheeler, N.

**Bldg. 224

MAGNETIC MATERIALS

Ldr: L. H. Bennett
Secy: J. M. Hill

Drew, R. V.
Mathews, D. E.
Shull, R. D.
Swartzendruber, L.

Students
Boomer, M.
Chen, T. A.
Fan, M.
Keisman, E.
Thorne, S. T.

Guest Scientists
Aviram, I.
Akinci, G.
Ettedgui, H.
Lundy, D.

METALLURGICAL PROCESSING

Ldr: J. R. Manning
Secy: S. L. Candore
Typist: M. E. Pickett

Bendersky, L. A.
Biancaniello, F. S.
Boettinger, W. J.
Boyer, P. A.
Burton, B. P.
Coriell, S. R.
DeWit, R.
Fields, R. J.
Gayle, F.
Handwerker, C. A.
Hardy, S. C.
Harne, D. E.
Jiggetts, R. D.
Low, S. R.
Parke, R. L.
Reno, R. C.
Ridder, S. D.
Schaefer, R. J.

Students
Huber, D. M.
Richards, E. L.
Williams, M. F.

Guest Scientists
Kattner, U.
Lee, D. J.
Shechtman, D.
Rhee, W. H.
Roitburd, A.
Yoon, D. N.

Consultant
Sekerka, R. F.

MECHANICAL PROPERTIES OF METALS

Ldr: J. H. Smith
Secy: J. A. Vaughan

Hicho, G. E.
Shapiro, A. J.
Shives, T. R.
Smith, L. C.

Guest Scientists
Mallen, A. J.
Fields, B. A.
King, R.

CORROSION

Ldr: D. E. Hall
Secy: J. A. Vaughan

Anderson, D. B.
Bertocci, U.
Escalante, E.
Fink, J. L.
Flanigan, C. D.
Fraker, A. C.
Harris, J. F.
Harris, J. S.
Harrison, S. A.
Interrante, C. G.
Lohr, D.
Ricker, R. E.
Stoudt, M. R.

Students
Coleman, V.
Mattamal, M. D.
McNair, O. B.
Patel, R.
Hong Yu, R.

Guest Scientists
Li, C. I.
Lichti, K.
Oka, T.

Research Associates
Karras, P.
Sturrock, C.

ADVANCED SENSING

Ldr: H.N.G. Wadley
Secy: A. Nashwinter

Clough, R. B.
Johnson, W. L.
Kahn, A. H.
Norton, S. J.
Pitchure, D.
Simmons, J. A.

Guest Scientists
Krasicka, E.
Rosen, M.

Research Associates
Mester, M.

* Bldg. 231

HIGH TEMPERATURE CHEMISTRY

Ldr: J. W. Hastie
Secy: F. M. Stanton

Bonnell, D.
Plante, E. R.
Schenck, P. K.
Sessoms, A. B.

Guest Scientists
Joseph, M.
Kowalska, M.
Zhao, J.
Zmbov, K.

Research Associate
Horton, W.

RESEARCH STAFF

Metallurgical Processing Group

Bendersky, Leonid A.	o Analytical transmission electron microscopy o Aluminum alloys; quasicrystals o Intermetallics for high temperature application
Biancaniello, Francis S.	o Inert gas atomization; metal powder processing and consolidation o Special alloy, composites, and quasicrystal preparation o Melt-spinning; rapid solidification
Boettinger, William J.	o Relation of alloy microstructures to processing conditions o High temperature alloys/intermetallics o Rapid solidification
Burton, Benjamin P.	o Thermodynamic modeling of alloy phase diagrams o Experiments on metal-oxide phase equilibria o Order-disorder and phase separation in alloy systems
Coriell, Sam R.	o Modeling of solidification processes o Interface stability o Convection and alloy segregation during solidification
deWit, Roland	o Fracture mechanics o Dislocation theory o Stereology
Fields, Richard J.	o Mechanical properties o High temperature materials o Quantitative metallography
Gayle, Frank W.	o Aluminum metallurgy; intermetallics o Transmission electron microscopy o Structure/property relationships
Handwerker, Carol A.	o Interface studies o Metal matrix composites o Diffusion-induced grain boundary migration

Low, III, Samuel R.	o Mechanical properties and testing o Laboratory computer systems programming o Automated test design
Hardy, Stephen C.	o Alloy coarsening o Surface tension measurements o Interface segregation
Manning, John R.	o Metals processing o Diffusion kinetics o Interface reactions
Ridder, Stephen D.	o Inert gas atomization; powder processing o Microparticle rapid solidification o Solidification dynamics
Schaefer, Robert J.	o Hot isostatic pressing of intermetallics o Solidification processes o Electron beam rapid solidification

Advanced Sensing Group

Clough, Roger B.	o Acoustic emission o Mechanical properties o Surface modification
Kahn, Arnold H.	o Eddy current modeling o Electromagnetic theory o Solid state physics
Johnson, Ward L.	o Ultrasonics o Solid state physics o Point defects in metals & semiconductors
Norton, Stephen J.	o Ultrasonic, Mossbauer and NMR imaging o Inverse modeling
Pitchure, David J.	o Digital electronics o Ultrasonic instruments o High temperature measurements
Simmons, John A.	o Dislocation theory o Acoustic emission o Inverse modeling
Wadley, Haydn N. G.	o Dislocations and fracture o Acoustic emission o Ultrasonics

Corrosion Group

Anderson, David B.	o Industrial corrosion testing o Corrosion data evaluation o Corrosion database development o Expert systems for corrosion control
Bertocci, Ugo	o Electrochemical measurements o Computer modeling o Passivity and pitting
Escalante, Edward	o Underground corrosion o Corrosion in concrete o Corrosion rate measurements
Fraker, Anna C.	o Titanium alloys o Corrosion processes o Transmission electron microscopy o Surgical implant metals
Hall, Dale E.	o Electrochemistry o Corrosion of advanced materials
Harris, Jonice S.	o Scanning electron microscopy o Corrosion measurements o Mechanical properties
Harrison, Steven A.	o Computer systems programming o Software engineering o Laboratory automation
Interrante, Charles G.	o Hydrogen embrittlement o Nuclear waste disposal o Environmental testing o Welding metallurgy
Ricker, Richard E.	o Environmental induced fracture (stress corrosion cracking and corrosion fatigue) o Hydrogen embrittlement o Aluminum alloys o Advanced materials (composites and intermetallics)
Stoudt, Mark R.	o Physical metallurgy o Corrosion engineering o Environmentally induced fracture

Electrodeposition Group

Beauchamp, Carlos R.	o Computer simulation of electrodeposition processes on moving fibers o Electrochemical measurements of kinetic parameters
Brown, Henrietta J.	o Coating thickness SRM development o Simultaneous thickness electropotential (STEP) SRM development
Claggett, Sandra W.	o Scanning electron microscopy o Metallographic specimen preparation o General electroplating
Johnson, Christian E.	o Ultra-black coatings o Electroless deposition process o Metallic glass alloy deposition o Microhardness SRM research o Chromium deposition o Pulsed alloy deposition
Kelley, David R.	o Microhardness SRM development o Dye penetrant SRM development o Precious metal electrodeposition o Plating on aluminum
Lashmore, David S.	o Electrochemical mechanisms of coating processes o Pulsed alloy deposition o Composition modulated alloy deposition o Properties and structure of electrodeposited coatings o Amorphous alloys o Transmission electron microscopy o Metal matrix composites
Mullen, Jasper L.	o Development of automated hardness testing o Electrochemical measurements for determining metal corrosion o Analytical spectroscopy
Stafford, Gery R.	o Electrochemical transients o Electrodeposition o Molten salt electrochemistry

Magnetic Materials Group

Bennett, Lawrence, H.	o Magnetic measurements o Magnetic thin films o High T_c superconductors o Topology of local environments o Alloy phase stability
Shull, Robert D.	o Nanocomposites o Magnetic susceptibility o Mossbauer effect o X-ray and neutron diffraction o Scanning electron microscopy
Swartzendruber, Lydon J.	o Magnetic susceptibility o Magnetic methods, NDE o Gamma-ray resonance spectroscopy o Barkhausen effect

High Temperature Materials Chemistry

Bonnell, David W.	o High temperature-pressure mass spectrometry o Computer modeling o Levitation calorimetry o Laser-induced vaporization mass spectrometry
Hastie, John W.	o High temperature chemistry of inorganic materials o Phase equilibria thermochemistry and solution models o High temperature-pressure mass spectrometry o Chemistry of combustion

Mechanical Properties of Metals

Hicho, George E.	o Mechanical metallurgy o Ferrous metallurgy o Failure analysis o Preparation and certification of standard reference materials
Shapiro, Alexander	o Analytical electron microscopy o X-ray microanalysis o Image analysis o Transmission electron microscopy

Shives, T. Robert	o Hardness test methods o Mechanical properties o Failure analysis
Smith, John H.	o Mechanical properties of materials o Fracture of materials o Structural integrity analysis

METALLURGICAL PROCESSING

John R. Manning

The properties of alloys depend on their processing history. This dependence arises because processing conditions determine the microstructures and composition distributions that are developed in an alloy; and these features in turn determine the alloy properties. The NIST work on metallurgical processing has as its objective the understanding of these processing-microstructure-property relations and the development of methods for measurement, prediction, and control of metallurgical processes.

Work on powder processing, effects of solidification velocities, and non-equilibrium interface processes has continued. In addition, during the past year, on-going activities related to the mechanical properties of alloys and their dependence on alloy microstructure were incorporated into the Metallurgy Division and into this group. The goal of this work is to relate processing to microstructures to properties. As a further addition, phase diagram activities concerned with development of advanced materials such as metal matrix composites and intermetallics are included as part of the present report. Process scientists and alloy designers need to know what alloy phases are possible in order to choose processing paths that will avoid undesirable phases and produce improved final products.

Collaborations continue with industry, universities and other government agencies. In cases where NIST has special processing facilities, such as those on rapid solidification, visiting scientists come to NIST to prepare special alloys. Studies on metal matrix composites, intermetallics and hot isostatic pressing are supported by the Office of Naval Research and the Defense Advanced Research Projects Agency. Studies of directional solidification and alloy coarsening are being sponsored by the National Aeronautics and Space Administration. Cooperation continues with the national steel program for new technology, and an NIST/industry consortium is developing techniques for intelligent processing during supersonic inert gas metal atomization using our SIGMA atomization system.

FY 88 Significant Accomplishments

- The Al(matrix)-SiC(fiber) metal matrix composite system was examined for phase stability in the presence of a liquid aluminum phase. Transmission electron microscopy measurements were made of intermediate phases formed at Al-SiC interfaces and interface roughness generated by diffusion and reaction. Calculations and measurements were made of the Al-Si-C phase diagram to relate these measurements to equilibrium and possible non-equilibrium phases.

- High speed holograms (exposure times of 20 ns) and high speed motion pictures (10,000 frames/s) were used to investigate the operative disruption mechanisms during inert gas atomization of Sn-Sb and stainless steel alloys. This is the first application of holography techniques to metal atomization.

- An NIST/Industrial consortium on Metal Powder Atomization was formed to study particle size control during inert gas atomization. Approximately twenty 10 Kg batches of 304 stainless steel were atomized to determine the characteristics of the Supersonic Inert Gas Metal Atomizer (SIGMA) as well as to test new in-situ particle size measurement techniques.

- The consolidation of metal powders into solid pieces by Hot Isostatic Pressing (HIP) has been studied for pure copper and for TiAl intermetallic compounds. The experimental results were compared to theoretical models. In each case, the models are useful but require further development. For example, HIP densification of TiAl is rapid at 1000°C but involves microstructural changes not included in available theoretical models.

- A theory was formulated to predict the composition, long-range order parameter, and interfacial undercooling during the rapid solidification of intermetallic compounds. The theory predicts the solidification velocity at which an ordered crystalline phase must grow to form disordered crystal.

- A thermodynamic model for ω-phase in Ti-alloys was developed which permitted the calculation of metastable equilibria with α-Ti phase in Ti-Nb, Ti-Mo and Ti-V. Ternary phase diagrams were calculated of Nb-Al-O for the high temperature oxidation program and Ti-Al-Nb for the intermetallics program.

- A precision elastic and plastic properties data base was established for a new, weldable high strength steel. The properties were measured in tension, compression, and shear. The novel steel studied here is presently making possible a new class of light weight naval vessels and has significant cost-saving potential in many commercial applications.

- Automated techniques were developed for the quantitative assessment of the inclusion content of steels. A stereological code was written to convert the two-dimensional data into three-dimensional information about the inclusions. This research provides a scientific basis for quantitatively linking processing to the properties of finished steel products.

- The coarsening of Sn particles in eutectic Pb-Sn liquid was measured while the samples were being slowly rotated to avoid sedimentation and agglomeration of particles. For low volume fraction solid samples the particles coarsen with a temporal law in agreement with that predicted by theory for the case of constant fluid flow velocity.

- The effect of anisotropic crystal-melt surface tension on the cellular interface morphologies and the resultant microsegregation patterns which occur during directional solidification has been calculated. For most metallic alloys, the calculation, which includes a weakly non-linear analysis, predicts a solute distribution corresponding to hexagonal nodes near the onset of cellular growth.

- A five-year program to characterize dynamic crack growth and arrest in pressure vessel steels has been successfully completed. Scientific achievements include evaluation of brittle crack inertia and kinetics of plastic zone formation. The data base is being used to confirm the integrity of pressure vessels in nuclear reactors.

Solid State Processes and Interfaces

J. R. Manning, C. A. Handwerker, S. C. Hardy, J. W. Cahn, J. D. Lee*, and W. H. Rhee**

* Guest Scientist - Chonnam National University, Kwangju, Korea
** Guest Scientist - Korea Advanced Institute of Science and Technology, Seoul, Korea

Solid state reactions, especially those at interfaces, are critical in determining the properties of metal matrix composites, surface modified alloys, and alloys undergoing coarsening. Topics that are important in the current NIST work in this area include surface energy effects and the effects of stresses generated by diffusion at reactive interfaces.

Thermodynamics and Kinetics of Reactions at Non-Equilibrium Interfaces - Interface reactions and interface structures produced by these reactions are critical in determining the mechanical properties of composite materials. Resistance of composites to fracture and crack propagation depends strongly on the degree of bonding, the amount of fiber pull-out, and the structure of the matrix-fiber interface. The production of interfaces with the "correct" degree of bonding, a stable interfacial bond, and a morphology that does not undermine the strength of the reinforcing material will be important in application of metal-matrix composites as high strength, high stiffness materials and also will be crucial for high-temperature ceramic-matrix composites.

Except under unusual circumstances, the matrix metal and the fiber or particulate reinforcements will not be in thermodynamic equilibrium when the two materials are first brought into contact. The system is generally also far from equilibrium even when the fibers are coated to limit diffusion and reaction at the matrix-fiber interface. Thus, there is a need to understand the thermodynamics and kinetics of composite interface reactions, the types of interface phases and textures that can develop, and ways of controlling interface properties. To this end, the goal of this work is to provide guidelines for the prediction and control of interface reactions in the production and use of composite materials. Three general topics, examined both theoretically and experimentally, are being investigated: (1) nucleation and surface energy effects at interfaces, since these will determine the structure and texture of the fiber-matrix interfaces, (2) reactions in the Al-SiC system, a model system for practical composites, and (3) stress effects accompanying diffusion at interfaces, since these stress effects can strongly influence composite interface morphology and stress adjacent along the interface.

A general theory for the development of nucleation textures has been developed giving the relationship between the orientations of second phase particles nucleated at an interface and the surface energies of these phases. This theory includes anisotropies in all interfacial energies, including the change in the crystal-substrate energy with misorientation. This theory demonstrates that the orientations of crystals nucleating on a planar substrate should depend on the equilibrium shape of the crystal and all anisotropies in the interfacial energies and that minimizing the energy barrier for nucleation leads to predictions of strong orientation textures that can change abruptly with small changes in nucleation conditions. This general theory is applicable both to phases nucleated at solid-liquid interfaces, such as occur during formation of composites by liquid infiltration or electrodeposition into a fiber preform, and also, with some modification, to phases nucleated at solid-solid interfaces.

In addition, transmission electron microscopy (TEM) measurements were made of interface reactions in Al-SiC, a model composite system, to evaluate the phases present, the defect population, including dislocations and twins, and the interface roughness generated by diffusion and reactions. Calculations and measurements were made of the Al-Si-C phase diagram to relate the TEM measurements to equilibrium and non-equilibrium phases in this ternary system. The phase diagram study indicates that the only carbide phase formed between SiC and Al at temperatures lower than approximately 1000°C is Al_4C_3. The interface roughness and the amount of interpenetration between SiC and Al_4C_3 were found to depend strongly on the initial microstructure of the SiC.

Diffusion at an interface generates large coherency stresses and strains when the lattice parameter is a function of composition. The effects of stresses generated by diffusion at liquid-solid interfaces have been demonstrated in experiments in ~~the Mo-Ni and the~~ Mo-Ni-Fe systems, in which stresses created by diffusion perturb originally planar interfaces. In these experiments, both deposition and dissolution reactions were generated as a result of the compositionally induced stresses present in the solid phase at Mo(solid)-Ni(liquid) interfaces. This combination of reactions produces an interfacial instability and large scale roughening of the interface. Mechanisms for the initiation of the instability have been developed based on the thermodynamics of stressed solids and the time evolution of the instability has been analyzed, from initiation to final flattening of the interface after stress relaxation occurs in the diffusion zone.

When the stresses caused by diffusion are high in a solid-liquid system, a grain size refinement of the original solid can occur either by the initiation of the instability followed by pinch-off of the precipitating regions or by nucleation of stress-free grains in a matrix stressed by diffusion followed by wetting of the newly created grain boundaries by liquid. Recrystallization/grain size refinement has the potential for producing small particle sizes that are difficult to attain by other methods as well as the potential for causing catastrophic dissolution of reinforcing materials in composites, even when the reinforcing material is a single crystal.

Ostwald Ripening of Solid-Liquid Mixtures - Alloy coarsening and Ostwald ripening are driven by the decrease in surface energy that can be achieved from, for example, the growth of large precipitates at the expense of smaller precipitates. Studies of the coarsening of Pb-Sn solid-liquid mixtures are being used as models to study this type of process under conditions where coarsening occurs rapidly enough for efficient measurement and under well-specified conditions which can be readily compared with theory.

Coarsening measurements in solid-liquid systems are normally made with high volume fraction solid samples where the development of a skeletal structure stabilizes the particles and prevents sedimentation driven by density differences. During this past year we have been developing a sample rotation technique which permits measurements in low volume fraction solid samples. In this technique, a disc-shaped sample is rotated in the vertical plane so that the particles describe circular orbits as they settle. Although the coarsening studies are complicated by the development of particle clusters, the average particle radius seems to increase initially with a temporal exponent of 1/2 as predicted for Stokes settling. At long coarsening times, most of the particles are agglomorated into clusters. The buoyancy forces on these clusters are so large they cannot describe orbits in the liquid but remain stationary as the liquid rotates past them. Although the fluid flow in the sample during rotation is not well understood, the average particle diameter increases with a temporal exponent of 3/8 in this case which is the result predicted by theory for constant flow velocity.

Powder Processing

S. D. Ridder, F. S. Biancaniello, R. J. Schaefer, R. L. Parke, P. A. Boyer

An NIST/Industrial consortium on powder processing was initiated and has begun a multi-disciplinary study on automation of inert gas atomization. Research groups from NIST aided by the financial and scientific assistance of Crucible Materials, General Electric, and Hoeganaes Corp. have combined to develop sensors and control systems needed to automate the atomization process. The NIST research groups include the Intelligent Control Systems Group of the Factory Automation Systems Division, Fluid Flow and High Temperature Reacting Flows Groups of the Chemical Process Metrology Division and the Metallurgical Processing Group of the Metallurgy Division. The Supersonic Inert Gas Metal Atomizer (SIGMA) in the Metals Processing Laboratory has been chosen as the development tool for this consortium. The project was divided into four subtasks, each to be completed concurrently. (1) In-situ particle size sensor development using Fraunhoffer diffraction has progressed to successfully monitor particle size at the exit region of the SIGMA facility. Current efforts are aimed at improving the performance of the computer used to transform diffracted light intensity information into particle size distribution and continuing modifications to the viewports used to monitor the powder flow. Assistance on this subtask has been provided by the High Temperature Reacting Flows Group. (2) Process modelling studies have commenced using a specially constructed supersonic flow test bench. This facility, designed and operated jointly by the Metallurgical Processing Group and the Fluid Flow Group, duplicates the high pressure plumbing used in the SIGMA system and provides

access to the supersonic gas jets for precise measurements of temperature and pressure profiles in the supersonic gas jets. Figure 1 shows the axial gas velocity and sound velocity distributions calculated from the static pressure, stagnation pressure, and stagnation temperatures measured on this test bench. These intrusive sensor studies have been augmented with non-intrusive photographic analysis of gas and gas-liquid flows. Schlieren and shadowgraph density wave imaging have been used to photograph the shock and expansion waves in both gas only and gas-water supersonic flow. In addition, twenty ns exposure holography and 10,000 fps cinematography were used to capture the actual disruption phenomena during water and metal atomization. The results of these and future flow studies will be used to improve atomization efficiency and to determine the best process control strategy. (3) Running concurrently with the previously mentioned sub-tasks is a process parameterization study which, when completed, will delineate the effect of each process variable on the final particle size distribution. Numerous atomization runs have been completed in a variety of metal alloys including Sn, Al, Cu, Fe, and Ni-based systems. (4) Researchers from the Intelligent Control Systems Group have been enlisted to aid in the construction of the process controller itself. A PC-based system has been chosen to ensure adaptability to other metal atomizers with minimal expense.

Hot Isostatic Pressing (HIP) is an important method for consolidating metal powders into solid parts. The powder is enclosed in a gas-tight can and heated in an argon-filled pressure vessel. In the past, the pressure, temperature, and time required to attain full densification of the powder have been determined mostly by expensive empirical tests. Now, studies of the kinetics of HIP processes being carried out in a joint project with the Advanced Sensing Group are leading the way toward much more sophisticated control of HIP operations.

Powder samples encapsulated in cylindrical cans have been subjected to a range of temperatures and pressures in a HIP system, and sensors developed by the Advanced Sensing Group have been used to determine their diameter as a function of time. The results are compared to the predictions of theoretical models of the powder densification process and are used to refine some of the parameters of the model or to indicate areas in which the theoretical model requires further development. The project, jointly sponsored by DARPA and NIST, is devoting attention specifically to titanium aluminides, which undergo complex microstructural changes during the consolidation process. The objective of the project is to obtain full densification of the powders while maintaining control over the microstructural changes. Experiments to date have indicated that the metastable phase content of rapidly solidified titanium aluminides transforms to a more equilibrated phase content at temperatures below those at which significant densification occurs.

<u>Microstructures and Properties of Steel</u>

R. J. Fields, R. deWit, S. R. Low, III, D. E. Harne, R. C. Reno, R. D. Jiggetts

<u>Quantitative Metallography of Steel Microstructures</u> - The utility of steel depends on its mechanical behavior, formability, and other performance

characteristics such as surface finish and paintability. These properties are determined by microstructures that result from processing. Quantitative modelling of processing effects on performance require quantitative assessments of microstructural characteristics. In this research, attention was focussed on measuring inclusions and on grain size and shape distributions. The research has progressed along two paths: (1) development of standardized preparation and (2-dimensional) measurement techniques for inclusions and grains, and (2) stereological methods for deducing the 3-dimensional nature of the microstructure from the 2-dimensional measurements. The objective of this research is to provide a theoretical and experimental basis for quantitative stereological analyses of steel microstructures prepared by standardized metallographic techniques. This work provides a quantitative means of relating processing parameters, microstructures, and the performance of finished steel products.

Standardized procedures for preparing and measuring inclusions have been established. A computerized, automated microscope has been programmed to measure and collect the pertinent data on inclusions from the prepared samples. Application of new stereological analysis techniques to an artificial 2-dimensional inclusion distribution including prolate and oblate spheroids was successful, and we have applied it to inclusions measured on planar sections of 304 stainless steel without difficulty. This work is now being extended to analyze grain size and shape distributions, i.e., space-filling particles as opposed to dispersed particles like inclusions. Experimentally, in collaboration with the Fracture and Deformation Division, studies have been initiated on the effect of thermomechanical processing (TMP) on austenite grain size and shape distribution.

<u>Wide-Plate Crack-Arrest Testing</u> - Crack arrest refers to the sudden stopping of a fast fracture. Crack velocities and arrest toughness must be known to prevent disastrous failure of large structures, such as nuclear pressure vessels. In work requested by the Nuclear Regulatory Commission, we have used new strain-gage techniques and a 53 MN (12 x 10^6 lbf) tensile testing machine to measure these values for some of the largest steel specimens ever tested. Very large specimens, typically 10 to 15 cm (4 to 6 inches) thick and a meter square, are required to avoid intractable complications due to reflected stress waves during the fracture event. Tests of 16 single-edge-crack tensile specimens in a thermal gradient have now been completed. We have obtained crack arrest data at 115°C above the nil-ductility temperature, the highest temperature at which arrest has been achieved.

Of the four tests completed in the past fiscal year, two were performed on as-received A533B steel and two on a steel that had been heat-treated to simulate radiation embrittlement. Results showed that brittle fracture could occur at temperatures above the onset of ductile behavior as determined by traditional impact testing. Furthermore, arrest occurred at extraordinarily high values of applied stress-intensity factor. Research continues on identifying the physical or atomistic toughening mechanisms that permit a sharp cleavage crack to support such high crack driving forces. In addition, the data base resulting from these tests is being compiled in a personal computer accessible form for convenient dissemination to collaborators and other interested parties.

Mechanical Properties of High-Strength Low Alloy Steel - Two new high-strength low-alloy (HSLA) steels have been developed recently by U.S. industry. These are both chemical modifications of ASTM A710 steel. One has a nominal yield strength of 80 ksi and the other 100 ksi. They are both extremely tough and weldable. We have established a precision data base for these two alloys to facilitate the comparison of these steels with existing alloys and to aid in the design of structures and components fabricated from them. Present and future applications of these alloys include naval vessels, off-shore structures, railroad tank cars, and other commercial applications where high strength, toughness, and weldability are required. The properties determined include the elastic, plastic, and fracture properties measured in tension, compression, and shear. Precision true stress-strain curves were also generated up to the point of instability (i.e., necking, barrelling, or buckling) in the above three loading modes.

Controlled Solidification

S. R. Coriell, R. J. Schaefer, and J. R. Manning

In this research, the fluid flow, solute segregation, and interface morphologies which occur during solidification are being studied. In collaboration with G. B. McFadden, R. F. Boisvert, and L. N. Brush of the NIST Center of Computing and Applied Mathematics, R. F. Sekerka of Carnegie-Mellon University, J. I. D. Alexander of the University of Alabama in Huntsville, A. A. Wheeler of the University of Bristol, and D. T. J. Hurle of the Royal Signals and Radar Establishment, convection and interface stability during alloy solidification have been analyzed and modelled. The resulting predictive models can be used to guide alloy designers in choosing processing conditions that will provide optimum properties of the solidified materials, for example, in providing homogeneous material needed for electronic applications and in predicting the type of alloy segregation expected during the continuous casting of steel. A theory of dendritic growth developed by Kurz, Trivedi, and colleagues [Acta Met. 34, 823-830 (1986)] for binary alloys has been extended to multicomponent alloys, including steel. The model describes the columnar (directional) growth of dendrites. The radius of the dendrite tip is obtained from the marginal wavelength for the stability of a planar interface growing at a given velocity with a specified temperature gradient in the liquid. The solute field in the vicinity of the dendrite tip is obtained from the Ivantsov solution for a parabolic dendrite. The theory predicts the radius of dendrite tip, the concentration of solutes at the dendrite tip, and the temperature of the dendrite tip as a function of the processing conditions. A computer program is being written to calculate these quantities for multicomponent alloys appropriate to the solidification of steel.

Morphological stability theory predicts the conditions for which a planar crystal-melt interface is unstable for directional solidification of a binary alloy at constant velocity. For conditions near the onset of instability, a three-dimensional weakly nonlinear analysis has been carried out to second order in the interface deformation taking into account the anisotropy of the crystal-melt surface tension. The results of this analysis allows the

prediction of interface morphologies and solute segregation patterns near the onset of cellular growth. Growth of a cubic crystal in the [001], [011], and [111] directions has been considered. For a typical metallic alloy with a solute distribution coefficient less than unity, these calculations indicate that the solute distribution pattern should correspond to hexagonal nodes rather than hexagonal cells.

During directional solidification of a binary alloy at constant velocity, buoyancy-driven flow may occur due to solute gradients generated by rejection of solute at the crystal-melt interface. Numerical calculations of the solute and fluid flow fields in the melt have been carried out using finite differences in a two-dimensional, time-dependent model that assumes a planar crystal-melt interface and allows time-dependent gravitational accelerations (for example, vibration of the sample). The container walls are rigid and perfectly insulating to solute. For constant gravitational accelerations, as the solutal Rayleigh number (dimensionless solute gradient) is varied, multiple steady-states and time-dependent states may occur. The bifurcation from the quiescent state may be subcritical or transcritical, depending on the aspect ratio of the container. The solute distribution in the solidified material is obtained as a function of processing conditions.

Linear stability analyses are being applied to problems associated with the morphology of the crystal-melt interface. The effect of a two-dimensional stagnation flow on double diffusive and morphological instability has been calculated. The flow stabilizes the system with respect to perturbations that are perpendicular to the flow; the double diffusive instability is significantly reduced by the flow. The role of buoyancy on morphological stability during directional solidification vertically upwards for the case of rejection of a heavier solute at the crystal-melt interface will be investigated. Preliminary results indicate that for certain alloys a long wavelength instability can occur.

Physical Metallurgy and Processing of Advanced Alloys

W. J. Boettinger, L. A. Bendersky, F. S. Biancaniello, B. P. Burton, J. W. Cahn, U. R. Kattner

The development of new alloys with significant improvements in strength, density, and operating temperature requires an examination of new processing methods and new classes of alloys not normally employed in conventional metallurgical practice. Research is directed toward the examination of processing paths involving precursor metastable phases to produce stable two-phase structures after decomposition. For example, the formation of ductile second phase particles in intermetallics has the potential of increasing the toughness of these materials. Since equilibrium phase diagrams suggest only limited opportunities for precipitation from supersaturated intermetallic phases by conventional heat treatment, the possibility of extending the range of solubility of intermetallics by rapid solidification is being examined. Similarly large volume fractions of small, hard dispersoids can be formed in aluminum-base alloys by the same approach. Principles required to manipulate alloy microstructure by these methods are being sought.

Intermetallic High Temperature Alloys - Because of their high melting temperatures and low densities, many intermetallic compounds show promise for a generation of advanced alloys which may span the gap between ductile metallic alloys and brittle ceramic materials. Recently the application of rapid solidification and other new processing techniques (such as plasma deposition) to intermetallic alloys has stimulated a renewed effort in the development of high temperature intermetallics for structural application. In this year's research at NIST, it was established that an extension of the composition range of intermetallic compounds by rapid solidification was only possible when the compound was not too strongly ordered; i.e., line compounds do not respond significantly to rapid solidification. A theory was developed to predict the composition, interface temperature and long-range order parameter of intermetallic compounds as a function of solidification rate using an analysis of the interdiffusion across the liquid solid interface between a liquid phase and a solid phase composed of two sublattices. With increasing interface velocity the theory predicts a transition from the solidification of a phase with equilibrium long-range order parameter and with equilibrium solute partitioning to the solidification of a disordered crystalline phase with the same composition as the liquid. The critical velocities required to effect these transitions depends on the free energy function of the solid phase and thus are closely related to the strength of ordering of the solid phase. Some results are shown in Figure 2.

The decomposition of metastable microstructures formed by rapid solidification have been studied in NiAl-Ni_2TiAl and NiTi-Ni_2TiAl eutectic alloys. Under ordinary processing conditions these alloys contain nearly equal volume fractions of a B2 phase based on NiTi or NiAl and a Heusler ($L2_1$) phase based on Ni_2TiAl. Following rapid solidification by melt spinning the alloy is metastable single phase Heusler. During heat treatment, the isotropic APB structure disappears and is replaced by an array of coherent cuboidal precipitates of the Heusler phase in the B2 matrix. This structure, which often has excellent mechanical properties, can not be made in this alloy without the rapid solidification processing. The effect of the coherency strain due to the 1.6% lattice parameter mismatch between the B2 and Heusler phases on the formation of the cuboid structure has also been detailed (L. A. Bendersky, P. W. Voorhees, W. J. Boettinger, and W. C. Johnson, Scripta Met. 22 (1988) 1029-1034).

Titanium aluminides (Ti_3Al and TiAl) containing Nb additions are currently being studied in many laboratories for potential high temperature service. These alloys may be processed conventionally or by rapid solidification either as a monolithic or a composite matrix material. However, the phase equilibria in the Ti-Al-Nb system are poorly understood. Microstructural studies have been conducted using TEM on heat treated alloys. The compositions surround the composition Ti_2AlNb. All of the alloys contain large volume fractions of B2 or related phases when cooled at ~400 K/min from 1400°C. According to their fine scale of microstructure and crystallographic features, the related phases are the product of solid state transformation during cooling. At 1400°C all alloys are in the B2 (or possibly BCC) phase field. The presence of BCC or B2 phases may have significant impact on the toughness of these alloys.

For Ti-37.5 at% Al-12.5 at% Nb, the large grains of B2 phase stabilized at 1400°C decomposed during cooling to a single phase consisting of small (~1 μm) domains of a phase with a hexagonal Bravais lattice (a = .42 nm, c = .58 nm). Four rotational variants, related to the cubic phase by $\langle 111\rangle_c \| [001]_H$ orientation relationship, were observed. Each domain itself consists of three variants of translational antiphase domains. This phase is related to the ω-phase found in the Ti-Nb binary system.
In order to provide a framework for the experimental results on Ti-Al-Nb alloys and interpolation or extrapolation of data to other temperatures, a calculation of the Ti-Al-Nb ternary phase diagram was undertaken using the THERMOCALC DATABANK system. Some results are shown in Figure 3. The stability ranges of the phases were adjusted to match the experimental data obtained above and at the University of Wisconsin by J. H. Perepezko and Y. A. Chang. In the calculations of the binary systems, which are required before the ternary can be calculated, the liquid and terminal phases were described as sub-regular solutions. The intermetallic compounds in the Nb-Al and Ti-Al were described with the Wagner-Schotky model, where compounds are considered to consist of different sublattices allowing substitutional solutions on each of these sublattices. The Ti-Nb binary was calculated by D. L. Moffat and U. R. Kattner (Met. Trans. A (1988)) and includes the metastable ω-phase which seems to appear in an ordered variant in the ternary system. For the calculation of the ternary Ti-Al-Nb system, all phases must be allowed to have ternary ranges of homogeneity, which means that Wagner-Schottky phases must be modeled as metastable phases for the two other binary systems. The Gibbs energies of these metastable phases have been adjusted to obtain a match of the calculated diagram with the experimental data. Results from this calculation were presented at the CALPHAD XVII Conference.

A limitation of the THERMOCALC approach to calculation of phase diagrams involving order-disorder transitions is the use of the sublattice model to represent the ordered phases. The cluster variation method (CVM), which includes crystallographic aspects of order, is being extended to ternary BCC ordered phases which occur in the Ni-Ti-Al and Ti-Al-Nb as well as other systems. A code has been written for the tetrahedron approximation in the ϵ-G mode. Thus phase relations between A2, B2, B32, DO_3, and $L2_1$ phases can be treated. Both short- and long-range interactions may be considered. In most cases it is sufficient to consider only first- and second-neighbor pairwise interactions, but there are exceptions. For example, in the pseudobinary NiAl-NiTi (and therefore the ternary Ni-Al-Ti) it is necessary to invoke either: (1) first- through third-neighbor pair interactions, or (2) long-range "elastic" contributions, to successfully model the observed subsolidus phase relations. Another application is the Fe-rich regions of the Fe-Al (iron-aluminide) and Fe-Si systems which must be treated as ternaries (Fe↑-Fe↓-Al and Fe↑-Fe↓-Si) in order to model magnetic effects.

Aluminum-base Alloys - Aluminum alloys containing large additions (~10%) of elements with limited (<3%) equilibrium solubility form one of the new classes of alloys made possible by rapid solidification. In previous research on Al-Fe-Si alloys, the possibility of forming microstructures with fine distribution of dispersoids was demonstrated. This year, the microstructural response and the sequence of phase transformations during thermal annealing was studied by means of transmission electron microscopy

and differential scanning calorimetry. First, the amorphous spherulites formed in the Al matrix by rapid solidification transform (at ~380°C) to the complex α(AlFeSi) cubic phase. This transformation could be promoted by the similarity in local structure (icosahedral clusters) of both phases. At higher temperature (~430°C), the cubic α(AlFeSi) phase transforms by ordering to a trigonal phase (two modifications, rhombohedral α' and trigonal α", were found). Only at higher temperature (~500°C) did phases known to be equilibrium (monoclinic $Al_{13}Fe_4$ and hexagonal α_H) replace the lower temperature phases.

Since the lower temperature phases have a distribution and size desirable for good mechanical properties, an attempt was undertaken to stabilize these phases by adding a fourth element. Addition of Mn was found to be successful. The Al-Fe-Mn-Si alloys retain a fine distribution of α(AlFeMnSi) phase particles even after very high temperature annealing (550°C ~0.9T_m Al). One of the quaternary alloys, Al-5Fe-5Mn-5Si (wt%) was successfully atomized using the NIST atomizer. Preliminary study of the powder microstructure shows the presence of desirable fine distribution of the α(AlFeMnSi) phase. Work on these alloys will continue.

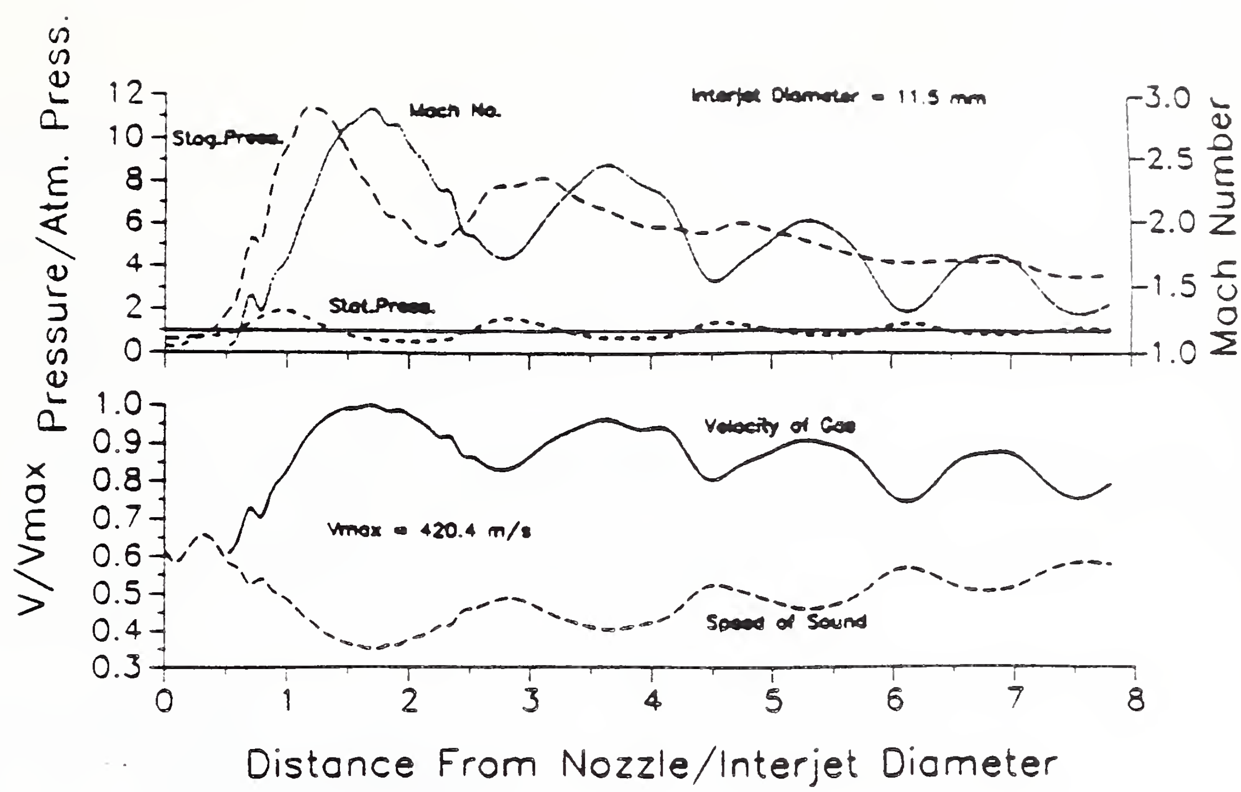


Figure 1. The upper plot shows the measured axial distributions of the static and stagnation pressure as well as Mach number produced by the die used in the SIGMA atomizer. These measured values were used to compute the velocity distributions shown in the lower plot. Note that gas velocities in excess of 400 m/s are achieved.

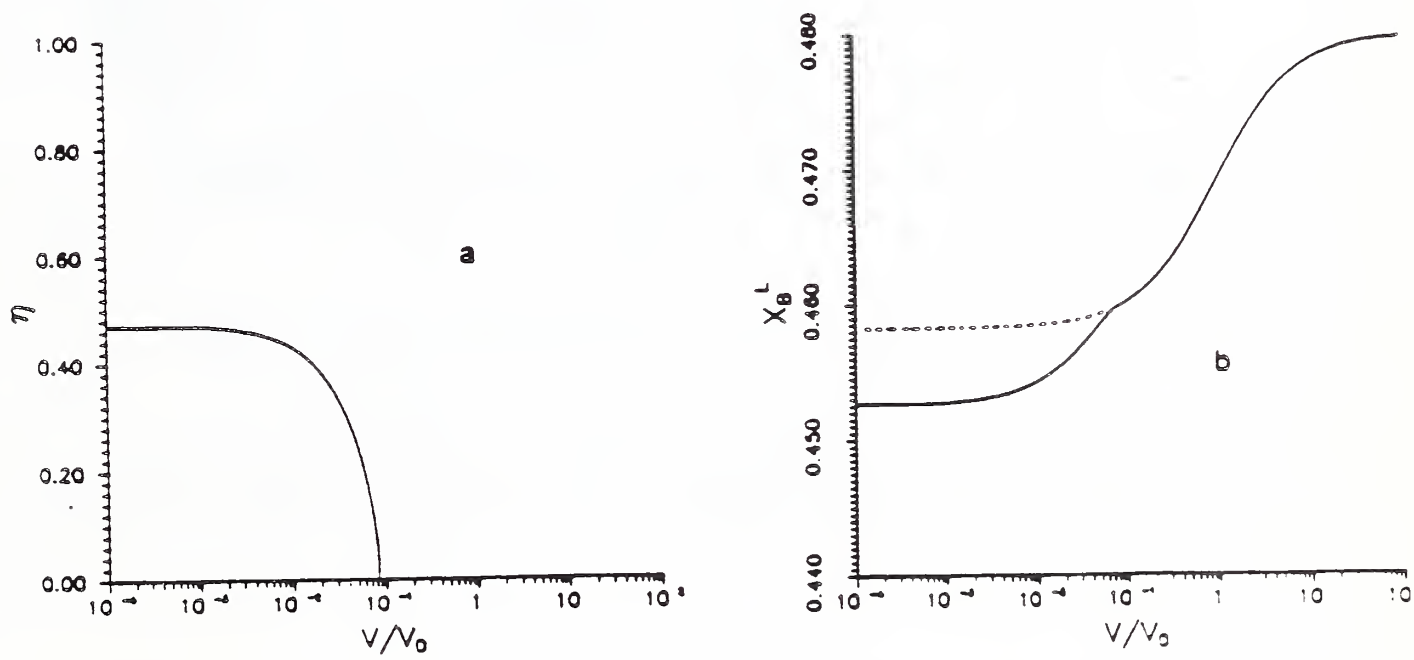


Figure 2. Prediction of non-equilibrium kinetic theory for the rapid solidification of an intermetallic compound. (a) Long range order parameter, η, as a function of dimensionless solidification velocity, V/V_D. Above $V/V_D \approx 10^{-1}$, only a disordered ($\eta = 0$) crystal can form. (b) Composition of the liquid, x_B^L, at the interface as a function of velocity, for a solid composition 0.48 when the congruent point of the intermetallic compound is 0.50. The dashed curve is an unstable solution of the theory at low velocity corresponding to $\eta = 0$.

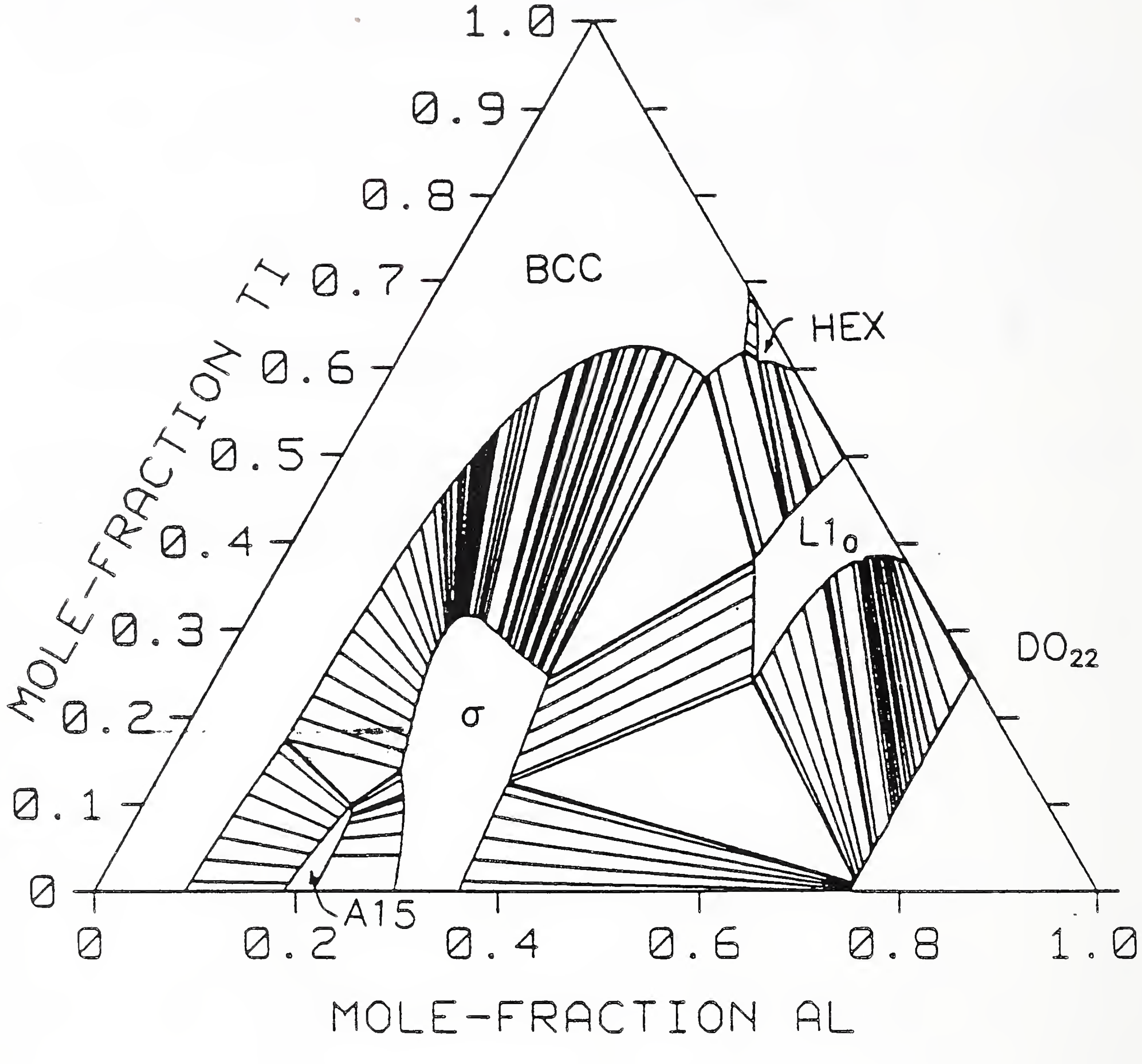


Figure 3. Preliminary calculation of the Ti-Al-Nb ternary system at 1400°C. The ternary phases are absent.

ADVANCED SENSING Haydn N. G. Wadley

The mission of the Advanced Sensing Group is to enhance the measurement science of noninvasive sensing methods and to apply them to the needs of materials science and engineering. These needs fall usually into two classes: methods for in situ observation of materials phenomena and techniques for sensing during process control.

The Group has aggressively moved into several research news areas this past year, while still maintaining its ongoing collaborative programs with the American Iron and Steel Institute and the Aluminum Association (this latter with partial support from the ONDE), and the research on interfaces in metal matrix composites.

Most visible of the new programs is a demonstration of DARPA's intelligent processing of materials (IPM) concept for the hot isostatic pressing of intermetallic composites. This DARPA funded program couples researchers in Metallurgy Division's Advanced Sensing and Processing Groups with the BDM Corporation, and the Universities of Cambridge (England) and Lulea (Sweden) to develop process sensing methods, validated process models and new control concepts incorporating artificial intelligence methods. Other important new work on process sensing has teamed the Advance Sensing Group with General Electric (Aircraft Engines Division) under NRL/DARPA/GE support to explore microstructure sensing during Rapid Solidification Plasma Deposition (RSPD). The Group has also been active in the new field of high Tc superconductivity where, with DARPA support, scoping studies of potential sensor methodologies have been initiated. Finally, the Applied and Computational Mathematics Program at DARPA has coupled NIST researchers in materials science with research mathematicians at Georgia Institute of Technology and the University of California at Los Angeles to model microstructure evolution using nonlinear methods emerging from the mathematics community.

Meanwhile, the ongoing collaborative programs with the AISI and Aluminum Association have each made substantial progress and several important milestones have been reached. The AISI/NIST program on internal temperature sensing has achieved the first imaging of a liquid-solid interface in a metal undergoing solidification. The ultrasonic technique used is an extension of the earlier developed method for measuring the internal temperature field in solid bodies. In related research, DOE funded researchers from Battelle Pacific Northwest Laboratory, Magnasonics, and AISI member companies have recently successfully tested the ultrasonic approach at ARMCO's specialty steel continuous caster at Baltimore. Thus, all the initial milestones for demonstrating feasibility have now been achieved. Future work looks toward the integration of this technique with process modeling of continuous casting.

The collaborative program with the Aluminum Association is exploring the use of electromagnetic (eddy current) methods to determine the temperature of aluminum during extrusion processing. Continued Aluminum Association support of Research Associate Michael Mester has had a major favorable impact on progress. An encircling coil technique has been successfully developed and

tested in an extrusion mill on bar and hollow sections. A new transmission technique is under test in the laboratory for use in other more complex geometries; it is scheduled to be evaluated during October 1988.

In the metal matrix composites area, we have completed development of an in situ test method for determining the strength of fibers and fiber/matrix interfaces in metal matrix composites. The technique utilizes acoustic emission to locate fiber fractures to ±100μm coupled with careful micromechanical measurements and a simple shear lag analysis. The technique is now being used to gain insight into the effect of process variables upon interface strength in aluminum-silicon carbide composites.

During FY 88 collaborations and interactions with industry, academia, and other government agencies have increased dramatically. Several cooperative programs have been started with General Electric and we are interacting with BDM, Pratt, and Whitney and Martin Marietta. New grants have been started with Prof. M. F. Ashby of Cambridge University (England), Prof. M. Fox and I. Hulthage at Carnegie Mellon University, Prof. Shui-Nee Chow at Georgia Institute of Technology and Prof. R. Chapman at George Mason University. An industry/academia consortium of researchers interested in hot isostatic pressing has been formed as part of the intelligent processing program. A Workshop on Modeling at the Greenbriar, West Virginia was co-organized with DARPA during September. Haydn Wadley now serves as Secretary and co-chairman of Program Planning Committee of the recently formed Materials Design and Manufacturing Division of AIME-TMS.

<u>FY 88 Significant Accomplishments</u>

- o An eddy current method for sensing metal temperature during extrusion processing has been developed. A simple encircling coil technique has been successfully evaluated in plant trials on simple shapes of circular or square cross section (solid and hollow). A two coil transmission technique has been tested in the laboratory for more complex geometries.

- o A sensor for determining density during hot isostatic pressing has been perfected and used to evaluate densificaiton mechanisms of $\alpha_2 + \gamma$ phase titanium aluminides. The data has been used to develop a HIP mechanism map for TiAl.

- o An ultrasonic methodology has been developed for mapping the liquid-solid interface in metals undergoing solidification. In aluminum, precision of $\pm$1 mm has been obtained.

- o A new test method for in situ determination of fiber and interface strengths in metal matrix composites has been perfected and used to explore the role of fiber coatings and liquid metal contact in aluminum-silicon carbide composites.

- o An ultrasonic study has been performed in the $YBa_2Cu_3O_{7-x}$ high T_c superconductor at cryogenic temperatures and large effects upon velocity and attenuation observed. These have been linked to a low temperature first order phase transition -- possibly magnetic in origin.

Design/Redesign of Process Pathways

Haydn N. G. Wadley, Mark Fox*, Ingemar Hulthage*, and Dan Backman**

* Carnegie Mellon University
** General Electric Company

Intelligent processing of materials concepts for many of the process steps used in powder metallurgy (see Figure 1) are now being explored. Common to each step is the development of experimentally validated predictive process models and knowledge bases and techniques (some based on Artificial Intelligence) for their interrogation. To date, it has been the intent to use this resource to control a single process step. In this DARPA funded effort, researchers at NIST, Carnegie Mellon's Robotics Institute, and General Electric are exploring the possibility of these process step simulators with emerging artificial intelligence methods for optimal design/redesign of a process path aimed to achieve a singular "goal state" combination of properties.

Interface Characterization in Metal-Matrix Composites

E. Drescher-Krasicka*, John A. Simmons, and Haydn N. G. Wadley

* Guest Scientist, Johns Hopkins University, Baltimore, MD

During melt processing of metal matrix composites opportunities arise for the formation of new phases at the metal-reinforcement interface. We are exploring the possible use of an ultrasonic method for observing the interface zone ("interphase"). In earlier work, our experiments identified the existence of interface waves at the cylindrical interface of a fiber reinforced composite. The analysis of the detailed structure of radial axial (leaky) modes in an isotropic clad rod has now been completed and shown to be in excellent agreement with these earlier experiments. An example of this agreement is illustrated in the Figure 2 showing the predicted and measured dispersion curves for a particular leaky mode in the Al/SiC system. We find the class of non-attenuating guided modes in a clad rod system is restricted to those materials where the shear modulus of the rod is less than that of the cladding. In all other systems a mode will leak energy from the rod into the cladding.

Over a large range of elastic parameters, the radical-axial wave modes in a clad rod system, which break down into four families (three of which are leaky or divergent), can be correlated with the modes of the bare elastic rod. In addition to these rod-correlated modes, the only other modes that were found were correlated to the limited number of "tunnel" modes in a cladding without a rod present.

We have found that the cylindrical geometry of the clad rod system produces a rich and dispersive structure for the leaky mode families and that, depending on the particular mode and frequency, analysis of the flow of energy reveals information about both the rod and the cladding.

This analysis was carried out using the detailed energy velocity field which generalizes the concept of group velocity. It was shown that by following the Poynting vector fields describing the energy flow, a lacuna can be expected after the startup of a leaky wave, and that the generator of this cone-like lacuna, after tracing a curved path near the interface due to the interference of the two components of the leaky wave, becomes a straight line inclined at some angle to the rod axis. Because of the interference pattern near the interface, the edge of the lacuna of a leaky mode will shift from its expected position estimated from the asymptotic leakage angle of the mode. This shift, analogous to similar shifts found in optics and in acoustics, has been shown to be obtainable directly by following the energy flow pattern. In some leaky waves, the energy pattern actually reverses itself and flows backwards; if this occurs asymptotically in the cladding, the leaky waves are backward leaking.

In addition to the complex patterns found in leaky waves, the energy velocity field displays the interference between plane and/or inhomogeneous waves near a plane interface. This pattern differs from the averaged energy flow, given by the group velocity, even in bare rods; and for clad rod systems possessing guided modes, the energy velocity at large distances from the interface is equal to the phase velocity of the cladding, not the group velocity of the mode.

If the structure near the interface zone is more complex, i.e. if the elastic constants vary with r and with frequency ω, most of the methods developed herein can, we believe, be extended to describe the leaky modes in that structure. Among the modes of such a system one can expect to find some which are sensitive to the elastic structure of the interface zone. Torsional sensitivity can be included by extending the above analysis to θ-dependent modes. In such cases the particle trajectories will no longer be planar ellipses but will follow slightly more complicated three dimensional paths. Anisotropy in the direction of the rod axis can also be included for the clad rod system, but more complex anisotropies can only be treated in the context of the planar interface using the type of formulation described in this study.

Test Methods for Interface Strength of Metal Matrix Composites

R. B. Clough, A. Manor*, Haydn N. G. Wadley and F. S. Biancaniello

* Guest Scientist, Armament Deveopment Authority, Israel

Acoustic emission and micromechanical techniques are being used to study the microdynamics of interfacial failure and fiber fracture in single crystal/single fiber Al/SiC metal matrix composites. These are Bridgman-grown in the form of dumbbell-shaped tensile specimens. Two different growth rates and SiC fibers (provided by AVCO-Textron) with either carbon-rich or

untreated surfaces are used to study the effects of processing path and fiber surface treatment on fiber and interfacial strengths. To do so, it is necessary to know the fiber segment lengths. Using conical piezoelectric sensors mounted at each end, the specimens are calibrated for emission source position using a pulsed Nd-YAG laser. Fiber and interfacial fractures are then located during tensile tests to within ± 100 μm, μ(1-10% of the fiber segment lengths) and subsequently verified by metallographic sectioning. Higher accuracies of ± 30 μm are obtainable by better noise-suppression techniques.

A micromechanical analysis has been developed, taking into account the mechanics of the test machine, to calculate the fiber strengths from the magnitudes of the load drops which occur on fiber fracture. A statistical model of fracture, combining a Weibull model that includes the tensile stress distribution from a shear lag analysis, has also been developed and used to characterize the fracture process. The results show that both fiber and interfacial strength always decrease with exposure time in the melt. The carbonizing surface treatment inhibits degradation of fiber strength by the melt through the rapid formation of a protective diffusion barrier of Al_4C_3. This barrier, at the same time, reduces the interfacial strength. The combination of high fiber and low interface strengths is generally thought to increase resistance to transverse cracking by first blunting the advancing crack tip with a shear microfracture at the fiber interface and then by increasing pull-out toughness by frictional sliding of the interface as the transverse crack passes through. Interfacial shear fractures were observed at the ends of fiber segments, which make these types of specimens also ideal for further testing of interfacial microfracture theories.

Eddy Current Sensing - Temperature Measurement of Aluminum Extrusion Processing

A. H. Kahn, M. L. Mester*, and Haydn N. G. Wadley

* Research Associate, The Aluminum Association

The joint project with the Aluminum Association for the development of eddy current sensing of electrical conductivity for the measurement of temperature has progressed with the design and construction of a through transmission sensor suitable for plant testing. In the previous year (1986-1987), a cylindrical two-coil surround system suitable for examining rods and tubes was designed, constructed, and tested successfully. The present design effort has been directed toward making the system applicable to the measurement of temperature in more complex extended shapes such as flatstrips, I-beams, and V-channels. These shapes all present cross-sections with appreciable regions which are flat and thin. These are heat tested by using transmit and receive coils placed on opposite sides o the flat region of the extrusion.

The electronic equipment is the same as that used for the surround coil system described in the previous annual report (NBSIR-87-3615; 59). The new coil system, fitted into a canister and ready to be installed in an extrusion

press, is shown in Figure 3. In this particular application two extruded I-beam shapes will pass horizontally through the two channels; the sensor will detect the temperature of the product in the upper channel only. The transient coil is located in the upper part of the channel; the receive coil is recessed in the horizontal web. Electrical leads and water cooling tubes are visible. The plant test will be carried out in the fourth quarters of 1988.

Modeling the Evolution of Intermetallic Microstructure

J. W. Cahn, John A. Simmons, Haydn N. G. Wadley, Shui-Nee Chow*, J. K. Hale* and S. Osher**

* Center for Dynamical Systems, Georgia Institute of Technology
** Department of Mathematics, UCLA

This new NIST/DARPA project addresses the application of emerging methods from the mathematical theory of dynamical systems to predict the evolution of microstructure in advanced intermetallic alloys. The goals of this program include:

- Solution of previously intractable materials science problems using new mathematical algorithms from computational algebraic topology, nonlinear partial differential equations and chaos theory.
- Development of new mathematical formalisms incorporating global stress and stochastic fluctuation-type variables to extend current reaction diffusion equations for multicomponent alloy systems.
- Development of image analysis techniques to characterize microstructure evolution.
- Design and implementation of experiments to critically evaluate theoretical models and their predictions.

The kinetics of the evolution of phases and their microstructural morphology occurring during solidification and subsequent annealing of metallic alloys are only qualitatively understood for multicomponent alloy systems. The first approximation to modeling this evolution will be carried out using the free energy function and generalized forms of the Cahn-Hilliard equation, but more advanced methods for determining the system propagator, such as path probability and transient time correlation methods will also be studied. Binary alloy systems containing titanium and aluminum, will be targeted and extension to ternaries containing nickel (or niobium) will also be investigated.

The crystallography of these ternary structures can be visualized in terms of four sublattices--each sublattice consisting of alternate sites on each of the two interpenetrating cubic sublattices making a bcc structure. Each of these four sublattices contains one-fourth of the atomic sites of the bcc structure. The simplest (Bragg-Williams) long range order model for this

system, leading to the reaction diffusion equation describing order ingredients, is then given by specifying the fraction of each of these sublattices occupied, respectively, by nickel, titanium and aluminum atoms--a total of twelve parameters, c_{ij}, the concentration of the i'th species on the j'th lattice. The free energy is then a functional of these twelve field and the temperature field. Knowing the (conserved) concentrations of nickel, titanium and aluminum atoms, together with the fraction of atoms on each of the fixed sublattices gives a total of 4 + 3 - 1 = 6 constraining relations leaving 6 (non-conserved) order quantities to describe the system. The reaction-diffusion equations governing this system can be derived in a similar fashion to the Cahn-Hilliard equation for systems with one ordering parameter and are of the form:

$$\partial c_{ij}/\partial t = D_{ijkl}\Delta c_k + f_{ij}(c)$$

where f(c) describes the cross-linking (or reaction) of species between sublattices and D_{ijkl} is an interdiffusion coefficient. This approximation can be improved by adding vacancies as another species and making the interdiffusion coefficients dependent on the concentrations. Still more accurate models are obtained by considering short range clusters of atoms, but the propagators for these variables have not yet been converted to reaction diffusion form.

The Cahn-Hilliard equations will be converted to ordinary differential equations using the method of inertial manifolds and the topological dependence of the solution forms on the initial conditions will be studied by Professors Chow and Hale. Computer models for describing microstructural evolution will then be developed in cooperation with NIST and compared with experiments carried out at NIST. ("Rapid Solidification and Ordering of B2 and $L2_1$ Phases in the NiAl-NiTi Systems," W. J. Boettinger, L. A. Bendersky, F. S. Biancaniello, J. W. Cahn, Proceedings of RQ6, Montreal, August. 1987 to appear in Mat. Sci. and Eng., 1988). Analysis of microstructures obtained will be developed in collaboration with Professor Osher.

High T_c Superconductors

W. L. Johnson, E. Drescher-Krasicka* and Haydn N. G. Wadley

* Guest Scientist, Johns Hopkins University, Baltimore, MD

Two studies of high T_c superconductors are underway in the group. A study of low-temperature ultrasonic elastic properties has been performed on the high-T_c superconductor $YBa_2Cu_3O_{7-x}$, with the aim of contributing fundamental data on electron/lattice coupling. Changes in ultrasonic velocity normally observed at the onset of conventional superconductivity are not apparent in this material due to much larger anomalous changes between 40K and 220K which, we discover, depend systematically on thermal history. These large velocity effects, and an associated attenuation peak, are found to be consistent with a first-order phase transition, perhaps magnetic in origin, occurring below T_c. Another DARPA project has been initiated to determine the feasibility of using ultrasonic and eddy current measurements as in situ sensors during processing of the high-T_c superconductor Bi-Sr-Ca-Cu-O. The

sensors are to be incorporated during heating and compaction of the ceramic powder in a hot-isostatic pressing (HIP) chamber. The eddy current sensor is currently being used in the HIP chamber and has the capability of monitoring electrical conductivity, volume changing phase transitions and powder densification with a resolution of one part in 10^4. Initial tests of the ultrasonic sensor indicate that reliable measurements of velocity can be made up to 650° and be possibly up to 1000°C, enabling determination of phase content and grain size/texture during sintering with or without the assistance of pressure.

RSPD of Titanium Aluminides

W. L. Johnson, E. Drescher-Krasicka*, A. H. Kahn, and Haydn N. G. Wadley

* Guest Scientist, Johns Hopkins University, Baltimore, MD

This recently initiated DARPA/NRL funded project teams NIST with General Electric (Aircraft Engines Division) and Drexel University to explore innovative control concepts for the Rapid Solidification Plasma Deposition of Intermetallic Composites. Work at NIST is directed at exploring potential methods for characterizing microstructure during the spray process. Both ultrasonic and eddy current methods are being investigated. Phase content, grain size and film thickness are the primary parameters of interest. An ultrasonic study has been initiated on a series of binary Ti-Al alloys with the purpose of determining velocity as a function of temperature in the α, α_2, β and γ phases.

Sensing and Process Modelling of Hot Isostatic Pressing

A. H. Kahn, R. J. Schaefer, R. Clough, W. L. Johnson, E. Drescher-Krasicka*, M. F. Ashby**, Y. Geffen***, M. L. Mester****, and Haydn N. G. Wadley

* Guest Scientist, Johns Hopkins University, Baltimore, MD
** University of Cambridge, England
*** Nuclear Research Center, Israel
**** Research Associate, The Aluminum Association, Inc.

Traditional control concepts for hot isostatic pressing are based upon sensing and maintaining temperature and pressure on a predetermined path, Figure 4. Trial and error is used to identify the "best" path. Recent advances in modeling the HIPing process at the University of Cambridge are being coupled with research on novel sensors for measuring density, temperature gradients, microstructure coarsening and microcracking to explore the feasibility of implementing an Intelligent Processing of Materials control concept, Figure 4. The DARPA funded program teams NIST researchers with the BDM Corporation, the University of Cambridge (England), and possibly Lulea University (Sweden) to test the IPM concept on the consolidation of intermetallic composites. A consortium of HIP utilizing organizations has been formed and helps guide the research and hasten industrial exploitation of emerging technologies.

The eddy current sensor used for measuring the electrical conductivity and temperature of aluminum extrusions also has the capability of being used for determining dimensions (and thus, density) during HIPing. This provides a basis for direct control of density itself rather than temperature and pressure which are not always capable of uniquely defining density. Research on titanium-aluminum alloy powders has necessitated construction of a new coil system capable of surviving at temperatures and pressures above 1000°C. The new coil system is wound on machined tubes of boron nitride; the wires are of platinum and platinum-rhodium alloys. Figure 5 shows the boron nitride based coils placed in the HIP furnace. The test sample consists of a copper or titanium tube with copper or titanium alloy powders sealed within, Figure 6. The system permits an in situ measurement of the cross-sectional area of the test sample during the processing.

Using data acquired with the above system we have computed the density as a function of time during a series of HIP runs. The histories of a typical densification runs on a copper powder sample is shown in Figure 7. The temperature and pressure schedules are displayed with the densities. The dependencies of rate of densification on temperature are clearly demonstrated. Detectability of subtler effects such as thermal expansion and contraction at the beginning and end of the process is possible when one examines the diameters corresponding to the measured cross-sectional areas.

This densification measurement is being used to explore the validity of HIP process models (University of Cambridge), Figure 8, and shape change models (Lulea University). The validated models are then being used in collaboration with BDM Corp. for numerical simulation of the densification process in order to determine the optimal change to a process path to achieve the "goal state".

Other sensors for measuring microstructure coarsening (grain size), temperature gradients (which control deleterious densification gradient phenomena and residual stress) and residual stress induced microcracking are being explored together with the development of appropriate models relating these quantities to temperature, pressure and time.

Steel Sensors

F. A. Mauer, D. Pitchure, S. J. Norton, Y. Grinberg*, and Haydn N. G. Wadley

* Guest Scientist, Atomic Energy Commission, Israel

This ongoing program is directed at satisfying steel industry needs for internal temperature sensing during the continuous casting and subsequent thermomechanical processing of steel. Previous work at NIST on solidified material has demonstrated the viability of an ultrasonic approach. Workers from Battelle's Pacific Northwest Laboratory and AISI members have subsequently demonstrated the viability of making these measurements in a steel mill using EMAT technology. NIST research during the past year has

focused on extending the ultrasonic approach to solidifying bodies where knowledge of internal temperature and the solid shell thickness during continuous casting could allow faster casting rates and reduced incidence of breakouts.

A fully automated ultrasonic temperature sensor developed at NIST in collaboration with scientists from the AISI was described in the annual report for 1987 and results from a test on a six inch square block of AISI 304 stainless steel were reported. In any application of this device to monitoring the continuous strand casting process the strand may still have a liquid center when it passes through the sensor. For this reason attention has been directed toward use of the NIST's ten-channel ultrasonic sensor for detecting and measuring this liquid core. Controlling the solid shell thickness while increasing the casting speed promises substantial improvement in the productivity of steel processing.

Initially, for laboratory tests, aluminum rather than steel has been used to simulate a steel strand in order to avoid the extreme temperatures associated with molten steel. A plate one inch think is used to simulate a section of a 6x6 inch strand in the apparatus shown in Figure 9. There is a two inch hole through the plate so that both the upper and lower chambers can be filled with molten aluminum when the stopper rod is raised. Suitable choice of the initial temperature of the plate and of the molten aluminum results in a portion of the material remaining liquid. By adjusting the power to the resistance heaters in the chamber walls, it is possible to control melting and solidification of the core for as many cycles as desired.

Measurements are carried out using laser pulses to generate ultrasonic waves on one side of the plate and an array of five piezoelectric sensors to receive them on the opposite side. The remaining two sides of the plate are similarly equipped so that time-of-flight data can be obtained on as many as fifty intersecting paths through the midplane of the plate in an identical fashion to the approach used for solid bodies in earlier work. (See Figure 10 for one set of ten ray paths used for some tests)

Because the heat flow, is greater on the two surfaces where the detecting transducers are located, the liquid-solid interface is elliptical. In order to reduce the number of measurements required and to simplify the analysis, we use a least squares technique to define an elliptically-shaped boundary with unknown center, major and minor axes. At least four measurements are needed to obtain the four parameters that define the ellipse. Using ten measurements, the best elliptical approximately to the boundary has been derived by fitting an ellipse to the time-of-flight measurements in a least-squares sense. The results for three different center temperatures are plotted in Figure 10 which shows the ten ultrasonic paths as well as the estimated outline of the liquid core. The results have been confirmed by embedded thermocouples and sectioning the block to determine the maximum extent of the liquid and by probing the interface with a ceramic rod during the experiment.

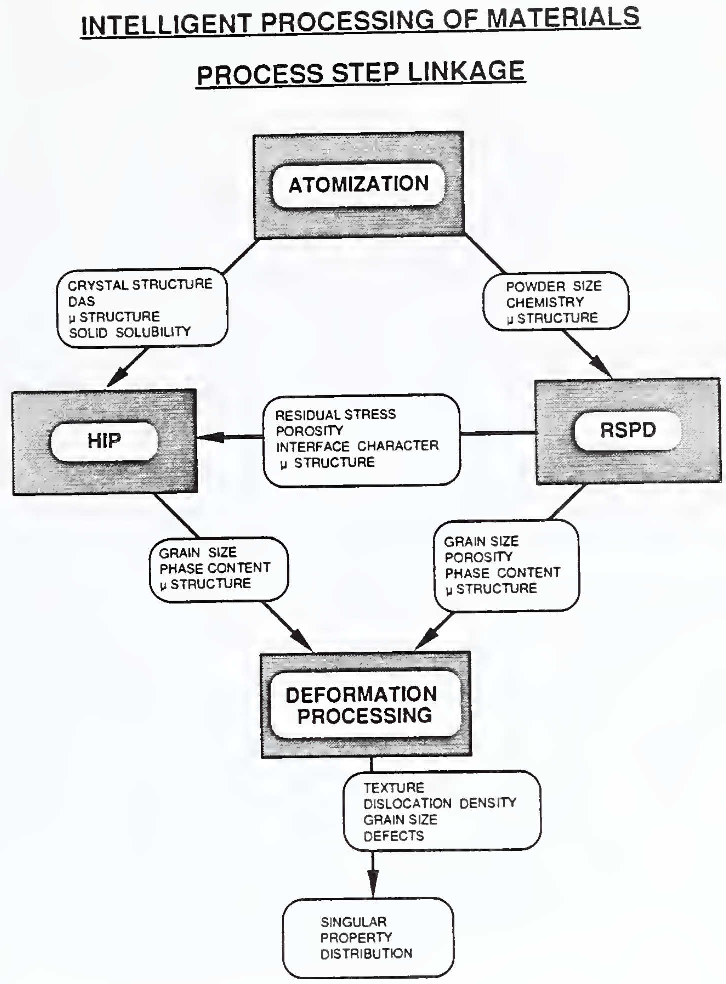


Figure 1. Schematic diagram showing the linkage of process steps to produce a component with powder metallurgy.

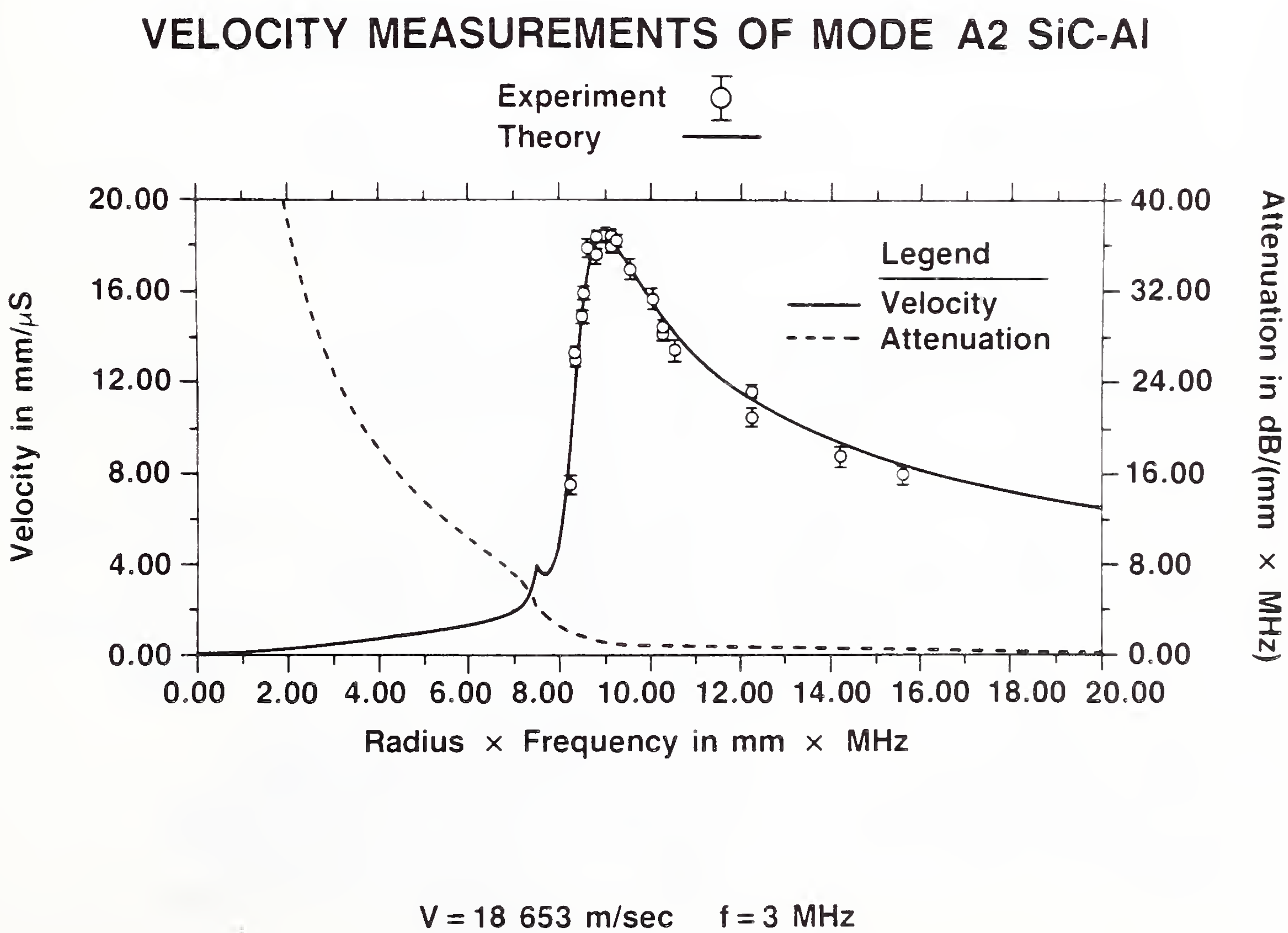


Figure 2. Comparison of theory and experiment for the A2 mode in specimen composed of a SiC rod in an aluminum matrix Al/SiC. The experimental apparatus was a prototype for an Interface Scanning Acoustic Microscope (IASM).

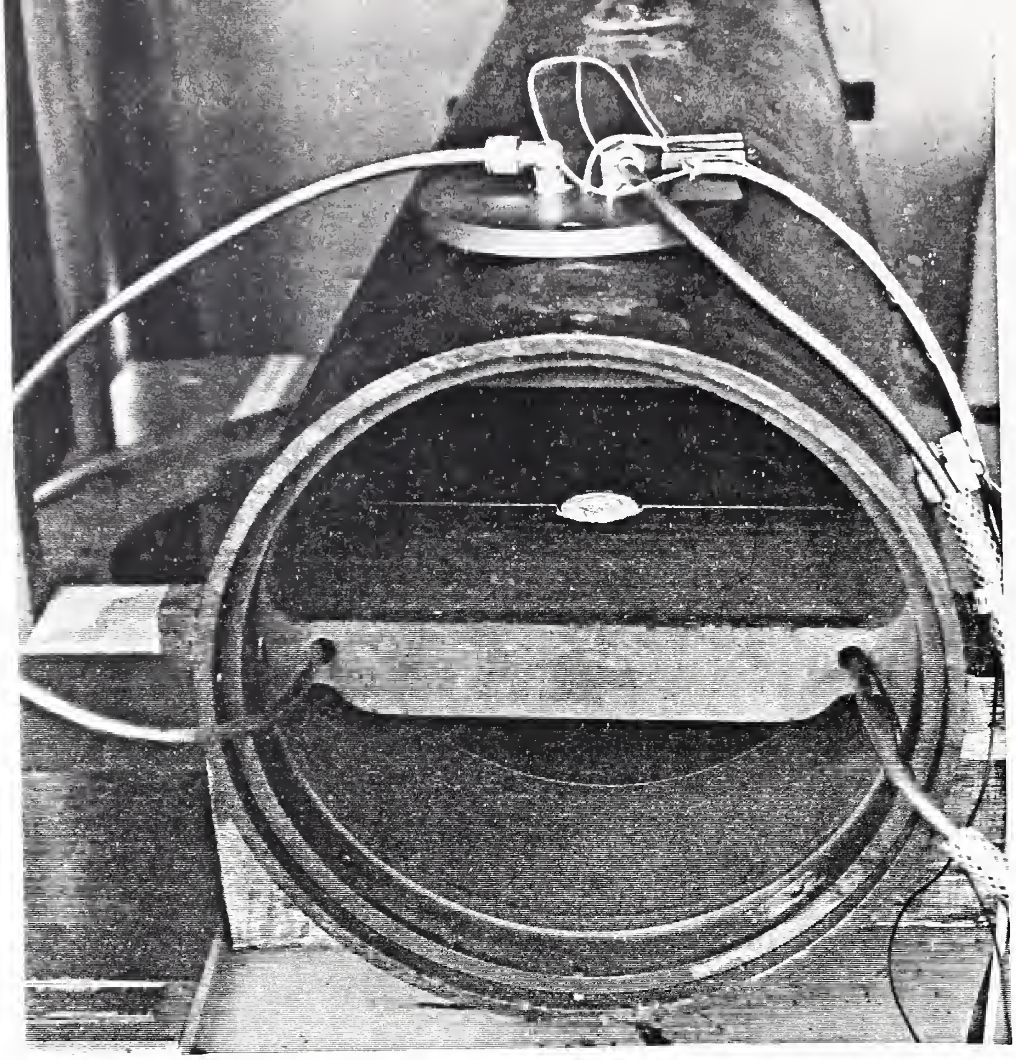


Figure 3. Transmit-receive coil system installed in canister, ready to be placed in an extrusion press for the measurement of temperature of aluminum products.

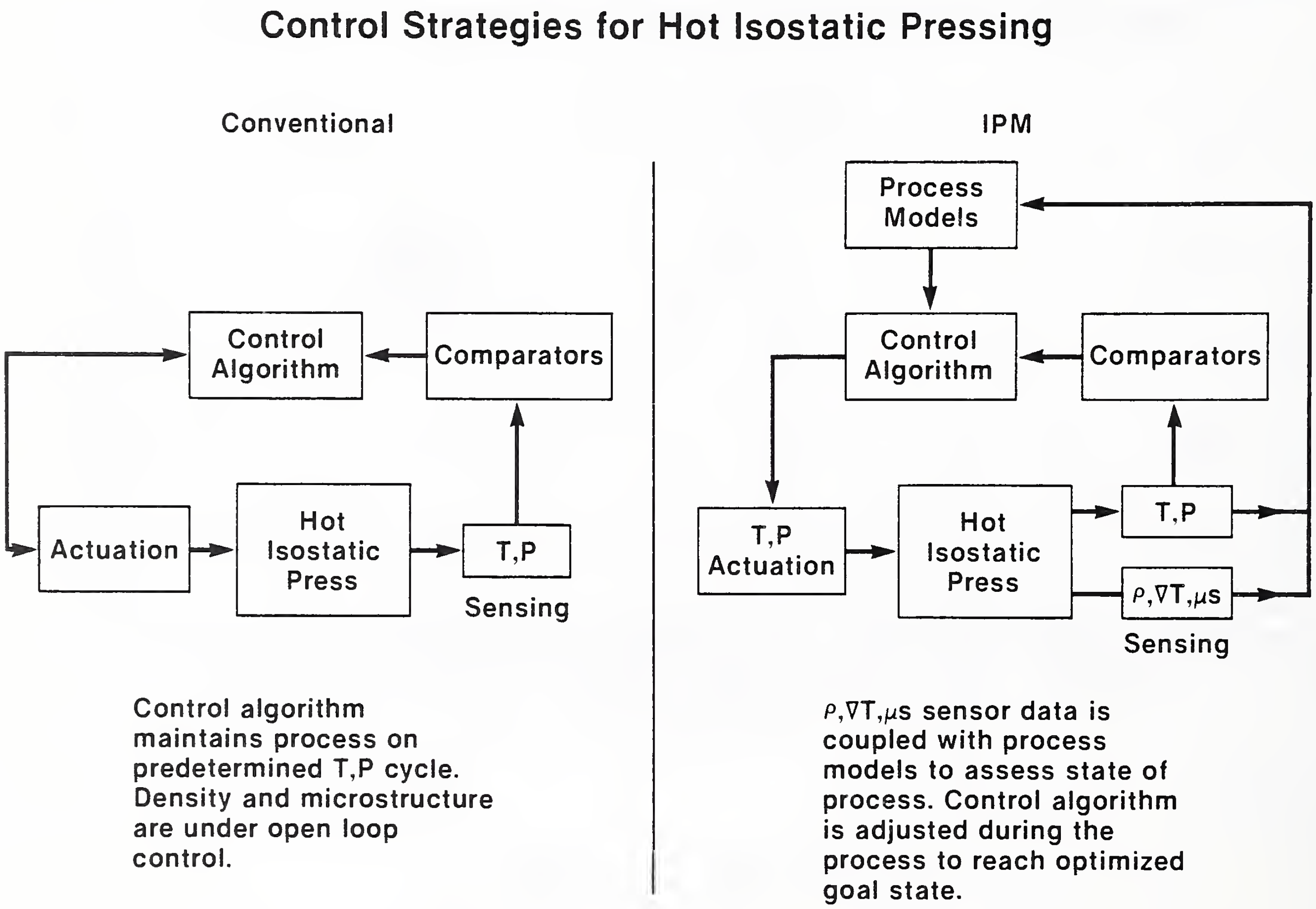


Figure 4. Conventional and Intelligent Processing of Materials strategies for control of hot isostatic pressing.

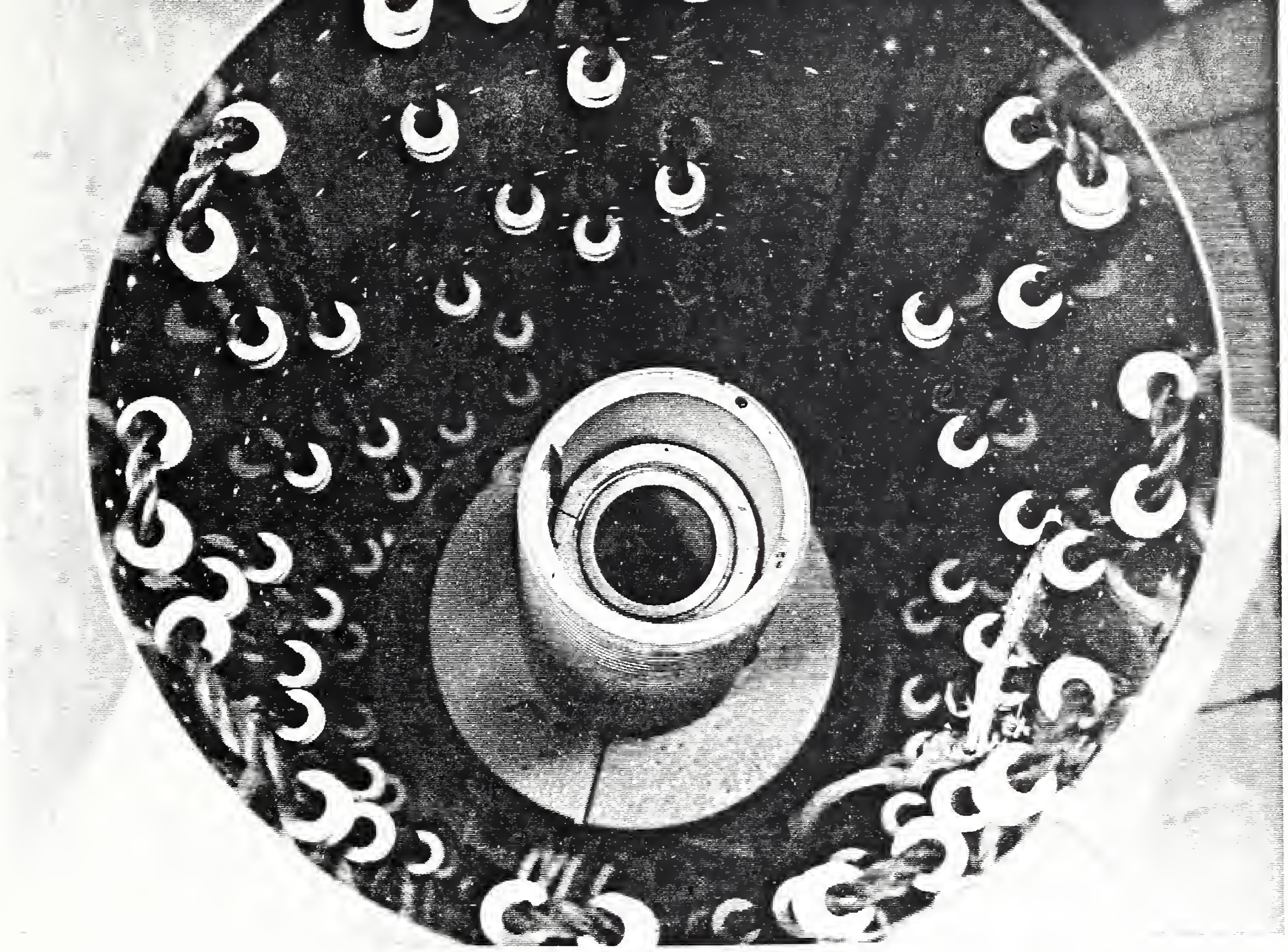


Figure 5. View of the boron nitride coil forms placed in the pressure furnace. The powders to be sintered are sealed in a metal tube and placed in the center of the coil system.

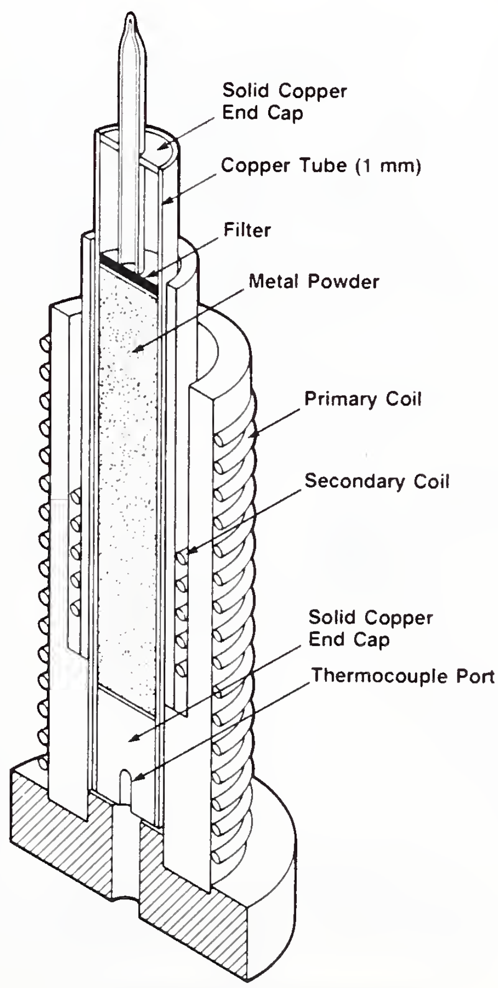


Figure 6. A schematic diagram of the HIP density sensor and a sample used for testing.

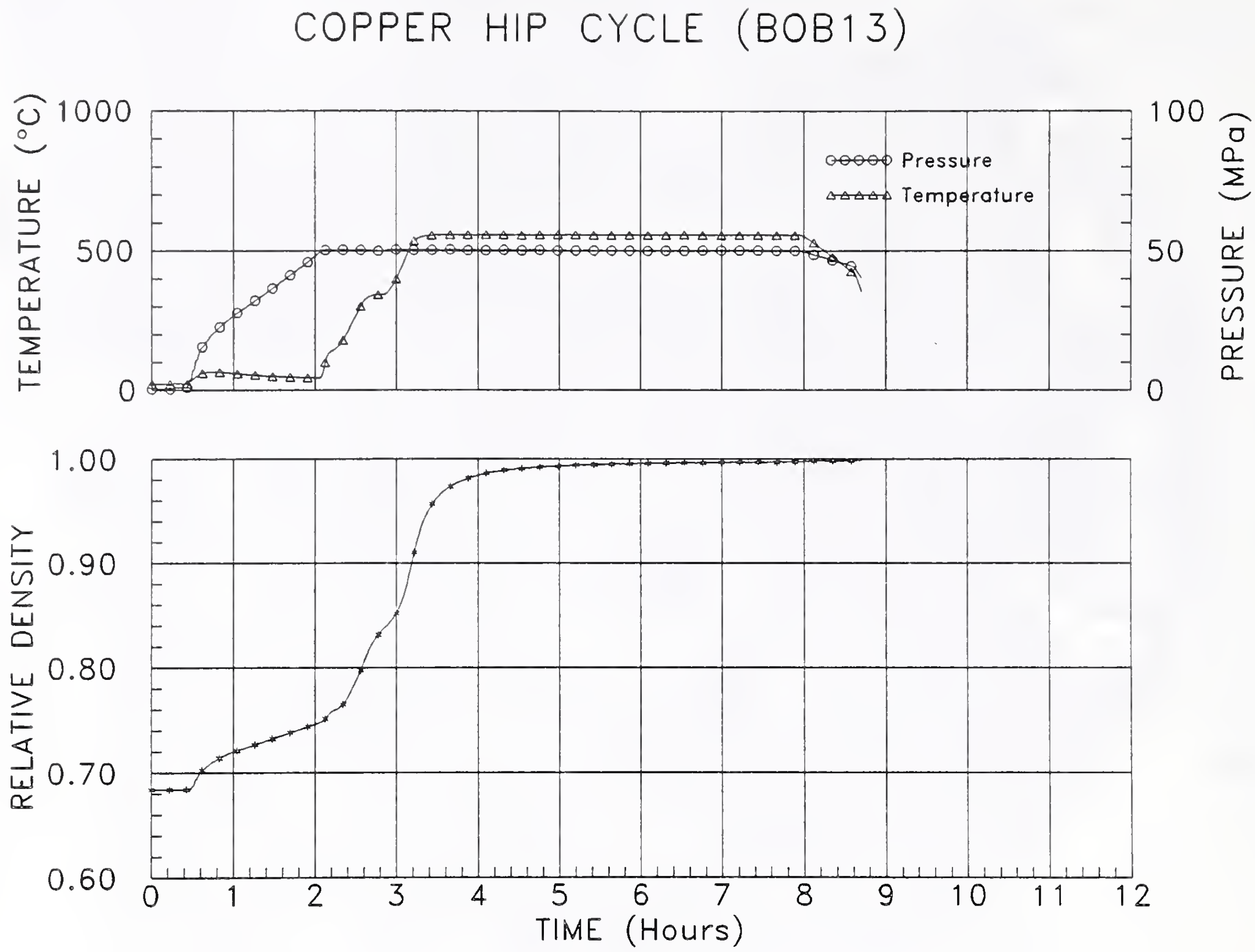


Figure 7. Relative density vs. time during HIP cycle #13. The temperature and pressure schedules are shown above the density data. Every tenth point is indicated with a marker.

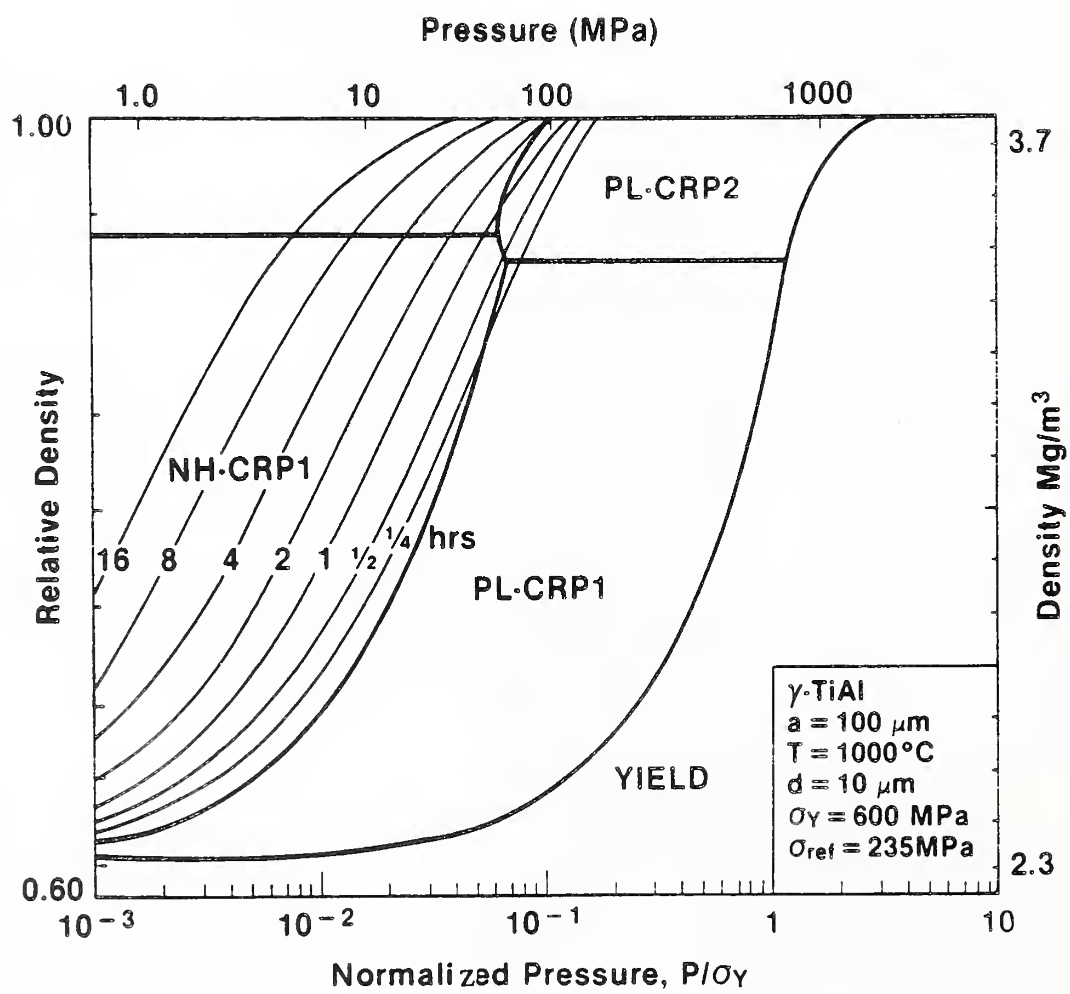


Figure 8. A preliminary HIP map for equi-atomic TiAl determined using the Ashby approach. The reference stress for power law creep (σref) has been adjusted to give a map that best matches experimental data.

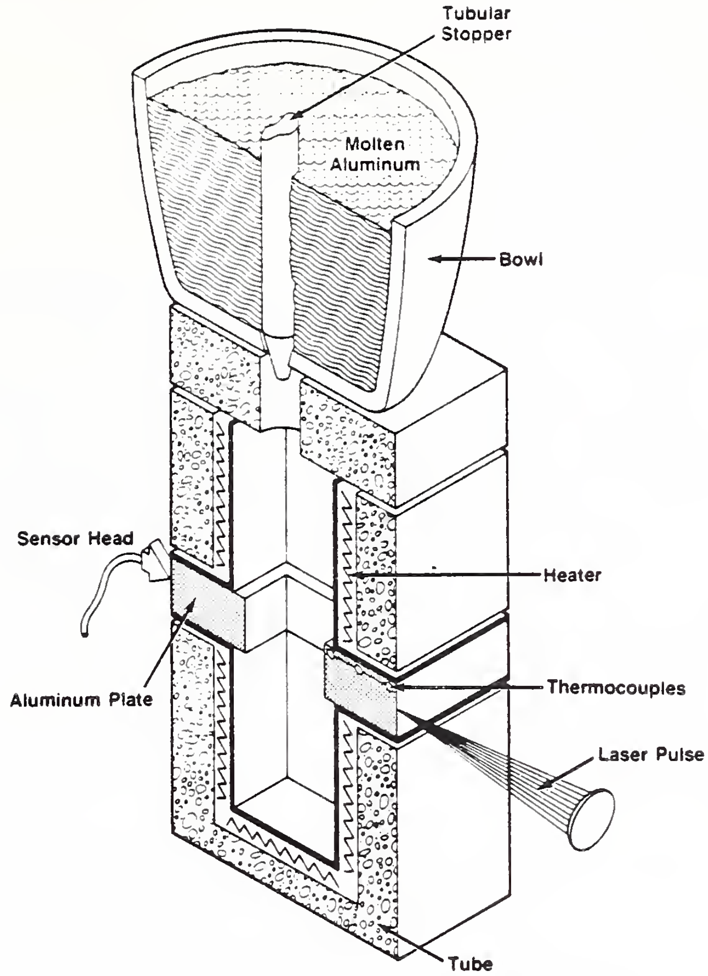


Figure 9. Apparatus for measuring ultrasonic time-of-flight through an alumunum plate with molten core.

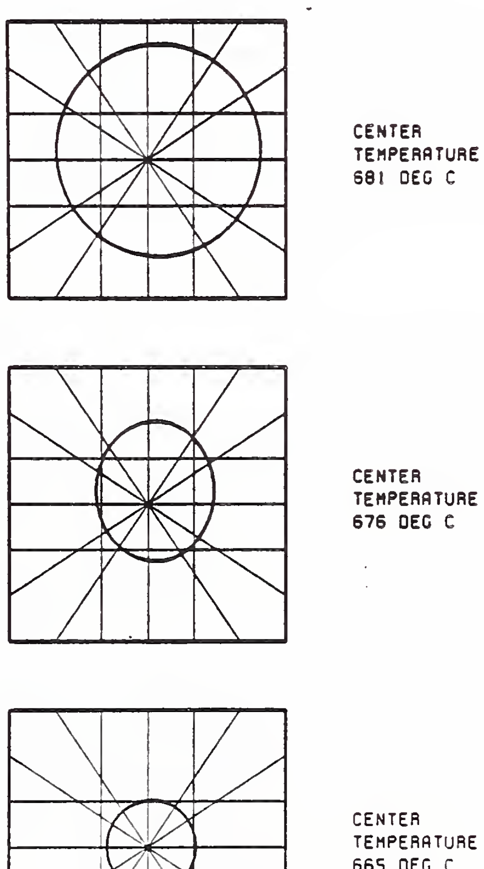


Figure 10. Computer reconstructions of the solid-liquid boundary at three distinct center temperatures.

CORROSION Dale E. Hall

Corrosion is a principal cause of shortened service life of metallic structures, and contributes to many catastrophic failures involving loss of life and property. The cost of corrosion to the U.S. economy is estimated to be close to $200 billion per year. The key factors in reducing these losses are better utilization of existing knowledge, better understanding of localized corrosion mechanisms which can lead to premature failures, and new research directed toward technologically significant materials and applications. The programs of the Corrosion Group address these areas.

In FY 88, programs were carried out in four main areas: (1) corrosion of advanced materials; (2) fundamental studies of localized corrosion mechanisms, stress corrosion cracking, and measurement methods; (3) corrosion data compilation, dissemination, and evaluation; and (4) corrosion problems in disposal of high level nuclear waste.

Research on the aqueous corrosion and stress corrosion cracking of advanced materials this year included a new program on the corrosion of ductile nickel aluminide and derivative alloys. These materials are being pursued for a number of low- and high-temperature applications, and are currently the focus of a technology transfer effort by Oak Ridge National Laboratory. Our corrosion research results have been shared with Oak Ridge and their industrial partners in a technology transfer workshop attended by potential producers, fabricators, and users of the alloys.

Progress was made in understanding the mechanisms of stress corrosion and localized corrosion, by combining experimental results with modeling studies. Studies of the mechanism of transgranular stress corrosion cracking in pure copper point toward the importance of localized dissolution as a common mechanism for stress corrosion cracking. Our understanding of pitting corrosion was advanced with the development of a stochastic model that qualitatively reproduces experimental current fluctuations associated with the formation of pits.

The Corrosion Data Program, a joint activity between the National Association of Corrosion Engineers and NIST, serves as a central source of reliable, evaluated corrosion data. The first commercially available software from the program has been enthusiastically received by users, and work is continuing on both an evaluated corrosion data base and the development of expert systems for use in materials selection and corrosion control.

Our assistance to the Nuclear Regulatory Commission includes both evaluation of published information and assessment of the technical issues involved in nuclear waste disposal. In addition, laboratory measurements have been made to increase our understanding of the influence of the environmental conditions in the repository, to determine the performance of candidate materials, and to develop more sensitive methods for detecting cracks in waste canister materials.

FY 88 Significant Accomplishments

- The NACE-NIST Corrosion Data Center initiated an industry-funded program to develop knowledge-base expert systems which incorporate symbolic representation of expert's experiences and logic in selection of materials and corrosion control methods for storage and handling of hazardous chemicals and downhole equipment for new oil and gas resource production. These systems are expected to become increasingly important information sources for corrosion scientists and engineers when dealing with critical materials applications.

- The aqueous corrosion behavior of ductile nickel aluminide was found to be controlled by nickel in a variety of environments. Stress corrosion cracking behavior of the nickel aluminide and a chromium-containing derivative alloy was shown to be consistent with a mechanism involving embrittlement due to hydrogen absorption.

- An experimental study of stress corrosion cracking of copper indicated that the formation of small pits in the oxide film, rather than fracture of an intact oxide film, was responsible for crack propagation. Comparison of the copper results with those of other face-centered materials suggests a common mechanism of transgranular stress corrosion cracking involving localized dissolution.

- An analysis of the fluctuation of electrical parameters, i.e. noise, accompanying localized corrosion has shown that frequency spectra of the noise can be used to determine the repassivation rate of metal surfaces. Computer simulations were used to show that the technique can provide useful results ~~even when large~~ numbers of difficult-to-resolve fluctuations are produced.

- Corrosion Group expertise in test methods for evaluating localized corrosion phenomena is being used to provide critical data for interpretation and evaluation of DOE-sponsored studies on corrosion of nuclear waste packaging materials. Key issues studied in the experimental program include the effects of projected repository conditions on corrosion behavior of carbon steel, pitting and stress corrosion cracking (SCC) of other corrosion resistant materials, and detection of SCC propagation.

Corrosion of Advanced Materials
D. E. Hall, R. E. Ricker, U. Bertocci, J. L. Fink

Corrosion of Intermetallic Compounds - Nickel aluminide is a new engineering material with a broad range of both high- and low-temperature potential applications, including jet engine parts, metal matrix composites, dies, and drill bits. The Corrosion Group work on nickel aluminide alloys is aimed at providing useful corrosion data and an understanding of the mechanisms underlying aqueous corrosion and stress corrosion cracking (SCC) behavior of the alloys.

Materials studied include the basic ductile alloy, IC-50, which contains a small amount of boron to provide ductility, and a chromium-containing version, IC-218, with superior high temperature oxidation resistance. Both cold-worked and annealed samples of each alloy were studied. The alloy specimens were supplied by Oak Ridge National Laboratory. For comparison, experiments were also performed on the parent metals, pure nickel and aluminum. The test environments were selected to provide a variety of corrosion behaviors: one parent metal (Ni) stable (0.5M NaOH); both parent metals passive without pitting (0.5M Na_2SO_4); both passive with pitting (0.5M NaCl); and, both parent metals unstable with the possibility of hydrogen effects (0.5M H_2SO_4). Slow strain rate experiments were conducted in the electrolytes and in air as reference, using a strain rate of 10^{-6}/sec.

The open circuit potential behavior of the IC-50 alloy was dominated by nickel in all electrolytes (Figure 1). Alloy and nickel potentials were not greatly affected by pH changes. Cyclic voltammetry, used to study the polarization behavior, indicated that the electrochemistry of nickel aluminide and nickel were essentially similar in all the electrolytes studied. Nickel dissolution was evident in acidic electrolyte. In neutral chloride, the current corresponding to pitting of Al was absent from the alloy voltammogram, and the alloy began to pit at about the same potential as nickel. In alkaline electrolyte (Figure 2), the peaks corresponding to the oxidation and reduction of nickel surface species were observed on both Ni and the alloy. The aqueous corrosion results indicate that, under a wide range of conditions, the known corrosion behavior of nickel is useful for predicting the behavior of nickel aluminide.

Slow strain rate experiments showed that both cold-worked and annealed IC-50 alloy experienced no stress corrosion cracking in the neutral and alkaline electrolytes. Loss of ultimate tensile strength was pronounced in acidic electrolyte, particularly with the annealed alloy. IC-218 alloy behaved similarly to IC-50. Fractography showed clearly that both the annealed and cold-worked alloy fractured transgranularly in a ductile manner in air. Similarly, failure was ductile in all electrolytes except acids, where failure was brittle and intergranular for both the annealed and cold-worked alloy.

The SCC behavior in acid indicated that a mechanism involving hydrogen embrittlement occurs at open circuit. In support for this mechanism, a plot of free corrosion potentials for the alloy in the environments studied showed that hydrogen generation is thermodynamically possible in acid but not in the other electrolytes.

Stress Corrosion Cracking of Aluminum-Lithium Alloys - Aluminum-Lithium alloys, combining higher strength and modulus of elasticity with lower density, are being evaluated for critical aerospace applications. Maintaining good fracture toughness and corrosion resistance in required product forms is a prerequisite for acceptance. Corrosion Group studies, in cooperation with Alcoa, are evaluating the role of grain boundary microstructure on stress corrosion cracking resistance of these alloys.

High purity Al-Li binary and Al-Li-Cu ternary alloys were given different heat treatments that resulted in different grain boundary structures but give

the same yield strengths (matrix precipitate size distribution). In contrast to normal aging heat treatments which result in different grain boundary structures and different yield strengths, this technique allows the evaluation of the influence of grain boundary microstructure on the stress corrosion susceptibility of these alloys without the confusion that results from simultaneously altering the yield strength. The results indicate that the grain boundary precipitates strongly influence the susceptibility of these alloys to stress corrosion cracking and that it may be possible to achieve maximum resistance to stress corrosion cracking at peak strength by adjusting heat treatment to control grain boundary microstructure.

Surgical Implant Metals

A. C. Fraker and J. S. Harris

The metals which are used in the human body as surgical implants for orthopedic, dental, heart pacemaker and other purposes include 316L stainless steel, a Co-Cr-Mo alloy, a Co-Ni-Cr alloy, a Ti-6Al-4V alloy and unalloyed titanium. The corrosion behavior and mechanical properties of the device are dependent on metallurgical aspects of the design and production. Corrosion behavior is important since metal ion release into the body should be avoided or kept at a low level. The device should not fail due to corrosion or to a combination of corrosion and mechanical property deterioration.

Repassivation Studies - Repassivation kinetics of surgical implant metal in simulated body fluids influence metal ion release into the body and also mechanical strength, which may involve surface film repair. Electrochemical data have been collected for determining repassivation rates and surface film stability. This involved exposing a clean metal surface to simulated body fluid at a preselected potential and measuring the current decay versus time at temperatures of 22°C, 37°C and 50°C.

Surface Films on Titanium and Ti-6Al-4V - Information on surface preparation was provided to various dental implant manufacturers. The success of dental implants had increased dramatically in recent years and now some dental surgeons report 85 to 95 percent of implants last for periods of 5 to 10 years. The materials used for most dental implants are titanium and Ti-6Al-4V. Dental surgeons receive the titanium implants in various conditions, including passivated, acid washed, grit blasted, porous coated, sterilized by various methods, etc. The chemical and metallurgical condition of the surface will vary with all of these treatments. The nature of the metal surface will influence reactions and forces of the tissue/substrate interface which determine whether the bone growth and firm attachment occur.

Electrochemical procedures for determining surface condition were described and demonstrated to the Artech Corporation, Falls Church, VA. This involved mounting the implant and measuring the open circuit electrode potential of the implant in a simulated body fluid. Such measurements can demonstrate whether the implant has been passivated, cleaned with an alkaline solution or exposed to other treatments. Oxide films on titanium materials progress through a series of Ti/O ratios to the highest oxide, TiO_2. The Ti oxide, and thus the electrode potential is dependent on chemical surface treatment, temperature, the environment, etc. Measured rest potentials in body fluids indicate that the Ti surface is an intermediate oxidation state. Determining

the optimum surface condition may be an important factor for increasing the dental implant success rate.

The effects of different heat treatments and small differences in composition on the mechanical properties of Ti-6Al-4V alloy were studied by comparison of the British and the U. S. extra low interstitial (ELI) grades of Ti-6Al-4V. Transmission electron microscopic structural analyses were conducted at NIST and were correlated with mechanical property measurements made by Catherine Asgian at Zimmer, USA of Warsaw, Indiana. These results indicated some similarities and some differences in the materials but showed there was no advantage in changing the American Society for Testing and Materials (ASTM) F 136 Specification for Ti-6Al-4V to include a higher oxygen content.

Corrosion Mechanisms and Measurement Methods

T. Cassagne, E. Pugh, R. Ricker, M. Stoudt, and J. Fink

Mechanisms of Stress Corrosion Cracking - Investigations of the mechanisms of transgranular stress corrosion cracking (T-SCC) of pure copper and brass have been carried out with emphasis on the film-induced cleavage model. According to this model, crack propagation occurs by discontinuous cleavage, each brittle event being initiated in a thin film formed at the crack tip by interaction with the environment. Dealloyed layers and oxide films have been suggested as a possible starting points for the cleavage event, but in the case of Cu, which is known to undergo T-SCC, selective dissolution as well as hydrogen embrittlement can be eliminated as possible causes. Thus the study focuses on the role of oxide films, which, in the first part of this work, were shown to be a prerequisite for the occurrence of T-SCC.

Experimentally, T-SCC of copper has been studied by conducting slow strain rate tests on single crystal specimens in aqueous sodium nitrite and sodium acetate. The tests were conducted at different pH values, electrode potentials and strain rates. Oxide formation was investigated by means of potentiostatic and potentiodynamic techniques on {110} single crystal faces, the reported fracture plane, in conjunction with ellipsometry and electron microscopy.

The oxide growth process was investigated in two solutions which induce T-SCC in copper, namely 0.1 M sodium acetate, pH 5.5 and 1 M sodium nitrite, pH 9. Initially a thin cuprous oxide film (10 Å or less) was found to form in both solutions. Subsequently, this film breaks down at multiple sites by pitting, and this is followed by the formation of a thick porous cuprous oxide (several hundred Å) by dissolution and precipitation.

In terms of the model, the thin oxide was expected to be the film nucleating cleavage. However, the results indicate that the formation of very small pits in the oxide film is responsible for crack propagation and not fracture of an intact oxide film as was originally suggested. Comparison of the behavior of copper with that of other face-centered-cubic materials suggests that a common mechanism of T-SCC involving localized dissolution is operative.

Experiments have been initiated to see if SCC of brass can be observed in chloride solutions. Because of aqueous complex formation, a significant part

of the electrochemical behavior is similar to that in ammonia solutions, where SCC is known to occur. Extensive dezincification is observed in the presence of oxidants, much less in solutions in equilibrium with respect to copper. Slow, constant strain rate fracture tests have so far failed to show T-SCC, however. Further studies at higher strain rates are planned.

<u>Corrosion Measurement Methods</u> - Fluctuations in the electrical parameters of electrodes undergoing localized corrosion contain information useful in understanding the nature of the corrosion process. Examples are the rapid current spikes often detected during the induction period preceding the formation of stable pits, such as those shown in Fig. 3a. The analysis of such fluctuations has been carried out by considering them as a form of shot noise. They have been analyzed both in the frequency- and in the time-domain. From the frequency spectra, such as those shown in Fig. 3b, the rate of repassivation can be obtained from the position of the "knee" in the spectrum. Comparison of the spectra of pure Fe and Fe-Cr alloys shows that repassivation in chromium steels is about one order of magnitude faster than in iron, as would be expected because of the greater corrosion resistance of the Cr-containing alloys.

Computer simulations have shown that the treatment is valid even when, because of the high number of spikes, they cannot be easily distinguished; this is the case in the simulation run illustrated in Fig. 4a. The frequency spectrum (Fig. 4b) is similar to those derived from the experimental data, and from it both the process intensity and its decay rate can be obtained.

<u>Corrosion Data Center</u>

D. B. Anderson, C. D. Flanigan, P. Karras*, C. P. Sturrock**, and K. A. Lichti***

* NACE Research Associate

** MTI-NACE Research Associate

*** Research Associate sponsored by the New Zealand Department of Scientific and Industrial Research and NACE

The Corrosion Data Center is a joint program between NIST and the National Association of Corrosion Engineers. The Center serves as a focal point for the development of personal computer programs to facilitate widespread distribution of evaluated corrosion performance and control data for engineering materials exposed to a wide range of industrial environments. Activities in 1988 centered on:

(1) Completion of the Cor-Sur™ corrosion data survey software, which provides isocorrosion charts for a variety of metallic and nonmetallic materials in over 1000 chemical environments. These programs have received strong industry acceptance, with over 600 copies sold to date.

(2) Leadership role in development of consensus corrosion data format standards through ASTM and NACE.

(3) Development of a prototype corrosion database structure and data entry from a large industrial corrosion data source. Arrangements have been completed to access additional data sources for inclusion

in this comprehensive program, which is designed to accommodate corrosion data from multiple sources in a standardized format.

(4) Initiation of two new industry sponsored projects which introduce the concepts of knowledge-base information processing to capture interpretive concepts of proven expertise. These expert system programs are expected to be milestones in our goal of developing innovative methods for distribution of corrosion information to a broad user audience.

The initial industry contract with the Materials Technology Institute of the Chemical and Process Industries (MTI) has helped establish the Data Center as a focal point for addressing the complex issues in gathering and disseminating evaluated corrosion data with an industry consensus on the relevance and applicability of the data. Additional contracts are currently under negotiation.

Corrosion Issues in Nuclear Waste Containment

C. G. Interrante, D. Anderson, U. Bertocci, E. Escalante, A. Fraker, D. Hall, J. Harris, S. Harrison, W. Liggett, E. Plante, E. N. Pugh, R. Ricker, J. Ruspi, M. Stoudt

Nuclear Waste Packaging Materials - In a program titled "Evaluation and Compilation of DOE Waste Package Test Data," initiated in FY85, NIST supports the High Level Waste (HLW) Management Program of the Nuclear Regulatory Commission (NRC) by evaluating the Department of Energy's activities on high level waste disposal. The waste packages, radioactive waste from nuclear power plants and other sources, are being designed for storage in a permanent repository. Potential problems threatening waste package integrity including pitting, stress corrosion cracking, and hydrogen embrittlement, are the focus of the Metallurgy Division's efforts. Another area of concern, leaching of borosilicate glass, is investigated with the assistance of consultants and NIST personnel outside the division. The program is supported by statistical and modeling specialists from the Center for Computing and Applied Mathematics, as well as by consultants from outside NIST. The HLW Data Center established at NIST includes over 986 papers and technical reports. A computerized data base provides access to these materials. The data base also includes the full texts of all technical reviews of published work.

The technical assistance rendered to the NRC includes formal reviews of the research and development efforts of the DOE Waste Package Program. Over 38 reviews were completed in FY88. Technical review of other related documents (from NRC, DOE and their contractors), interaction with the international technical community, and general review of all relevant present and planned activities of the DOE are also important elements of the program. Subjects considered in the review of published studies include expected modes of degradation, evaluation of various other failure modes, test methods, design of experiments, theoretical and modeling efforts, and laboratory testing. An important objective is to identify additional data and types of tests needed to demonstrate that the DOE waste package designs will meet NRC's performance objectives.

The materials research program provides data to aid in the interpretation and evaluation of data submitted by the Department of Energy (DOE) to the NRC for licensing the repository, materials and procedures for nuclear waste disposal. During this past year, DOE selected Yucca Mountain, Nevada as the first planned site for underground storage of nuclear waste. This site consists of tuff (crystallized volcanic rock) which has approximately twelve percent porosity. The rock contains very little water (2 to 3 mm flow/year) and oxygen is present. The selection of this site from among three candidate sites will sharpen the focus of future research. This year laboratory studies were carried out in four areas: (1) Effects of Transport and Resistivity on Corrosion of Waste Package Materials, (2) Pitting Corrosion of Steel Used for Nuclear Waste Storage, (3) Corrosion Behavior of Zircaloy Nuclear Fuel Cladding, and (4) Evaluation of Methods for Detection of Stress Corrosion Crack Propagation in Fracture Mechanics

<u>Effects of Transport and Resistivity on Corrosion</u> - Studies sponsored by DOE indicate that if the metals considered for nuclear waste containers corrode uniformly, their lifetimes will be thousands of years. If nonuniform corrosion such as pitting occurs, however, the lifetime of the containers will be reduced to a few hundred years, not enough time for the nuclear waste to lose its nuclear toxicity. This study is considering the effect of transport and resistivity properties of the environment on the form of corrosion that occurs on steel.

Small steel coupons were immersed in a matrix of environmental conditions in which transport and resistivity were controlled. Electrochemical measurements, which include corrosion rate measurements and measurements of oxygen transport through media simulating underground conditions, indicated that in a wet sand environment where transport of oxygen is relatively low, corrosion rate is also low, and in a sand-free solution with high oxygen transport, corrosion rate is high compared to the sand environment. In addition, a high conductivity environment results in a higher rate of corrosion for steel compared to lower conductivity environments. The results indicate that oxygen transport and electrical conductivity in the repository environment may have significant effects on corrosion of waste containers, and are important properties to consider in characterizing the repository environment.

<u>Pitting Corrosion of Steel Used for Nuclear Waste Storage</u> - Steels, such as AISI 1040, and ASTM A-27, grade 60-30, and others in the low to intermediate carbon range have been considered for use as nuclear waste canister materials or as borehole liners. Stainless steels, such as 304 and 316, also have been considered for use as canister materials. This project deals with localized corrosion behavior of steel with emphasis on A27 steel. Electrochemical measurements were made to determine susceptibility to pitting in a high pH (9.75) electrolyte containing various ions which could be present in repository environments. Cyclic polarization measurements of A27 steel at 22°C and 95°C, as described in ASTM G61, showed that the pitting potential was slightly positive to the corrosion potential and that pitting probably would occur on a large scale. Microstructural analysis showed the presence of shallow pits and preferentially corroded pearlitic regions. Corrosion

rates for A27 under these test conditions were 6 mils per year (mpy) at 22°C and 12 mpy at 95°C.

Corrosion Behavior of Zircaloy Nuclear Fuel Cladding - Approximately ninety percent of the nuclear waste to be stored will be in the form of nuclear fuel with a Zircaloy cladding. This project deals with studies of surface oxidation, passivity, and susceptibility to localized corrosion such as pitting or stress corrosion cracking (SCC) of the material. Zircaloy materials were obtained from Teledyne Wah Chang and cladding was obtained from the General Electric Co. and Babcock and Wilcox. An extensive literature review was conducted on the corrosion of Zircaloy, and an analysis of the published data was prepared and submitted to the NRC. Electrochemical tests in high pH aqueous media at 95°C indicate potential pitting problems, and further studies are being conducted.

Evaluation of Methods for Detection of Stress Corrosion Crack Propagation in Fracture Mechanics Specimens - The purpose of this project is to detect crack extension or crack initiation activity, resulting from stress at flaws or material discontinuities, which occur after long times of up to 4.5 years. This project is directed toward early detection and toward developing procedures for accelerated testing. An acoustic emmission technique is used for these measurements. The success of this technique would result in the capability to measure crack growth at a sensitivity which has not been possible previously and would improve predictions of stress corrosion cracking suceptibility and failure. Instrumentation and software were developed for these tests. Tests were conducted on 2.25 Cr - 1 Mo steel in deaerated acetic acid solution saturated with hydrogen sulfide. Some aspects of the test apparatus are being changed to keep the environment oxygen free. After the test method is developed, other environments and materials will be studied.

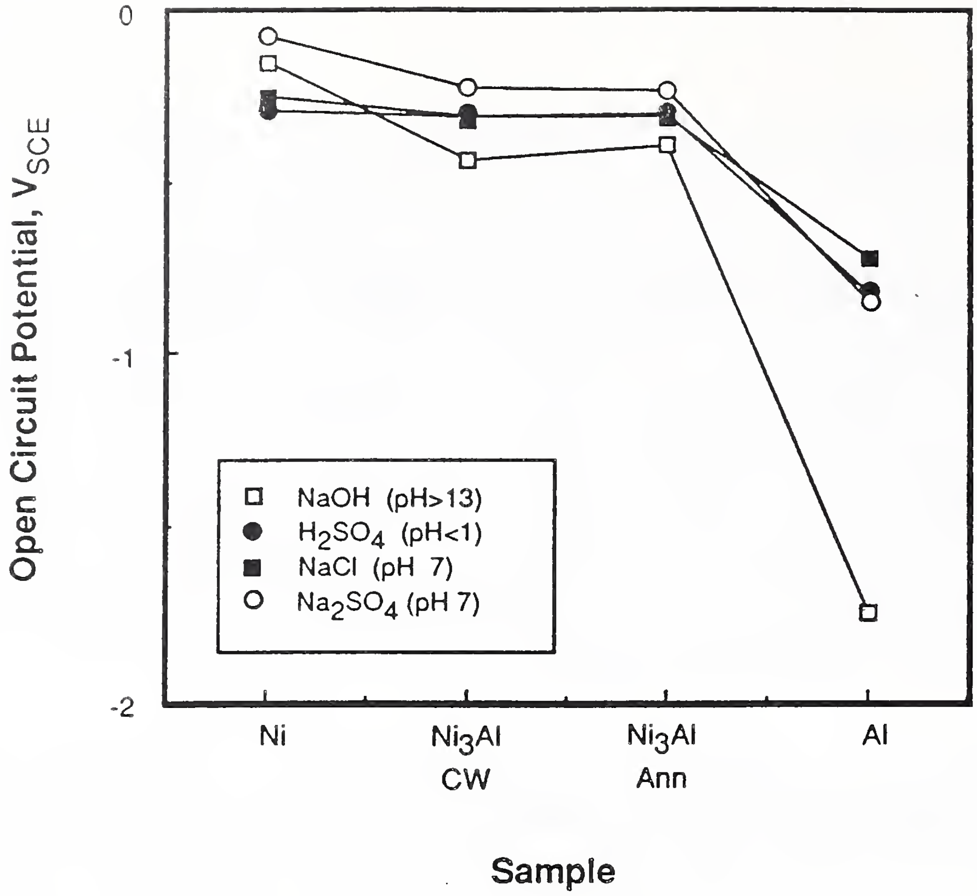


Figure 1. The open circuit (free corrosion) potentials of cold worked and annealed nickel aluminide are dominated by nickel in acidic, neutral, and alkaline electrolytes.

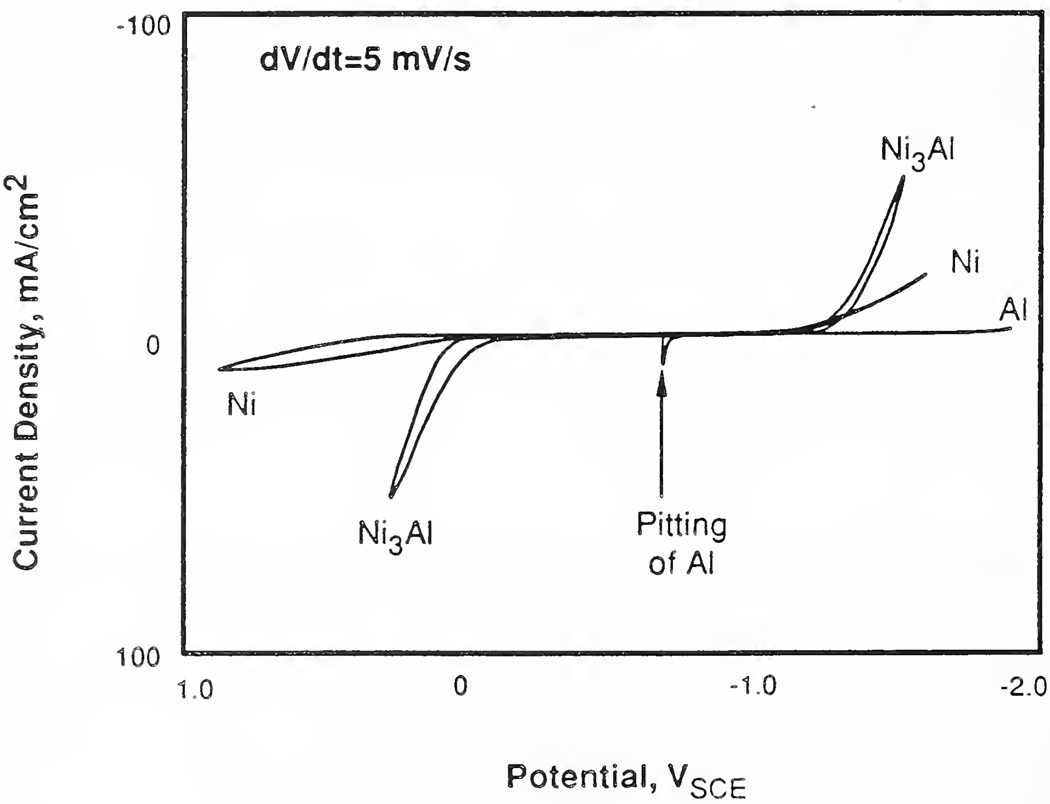
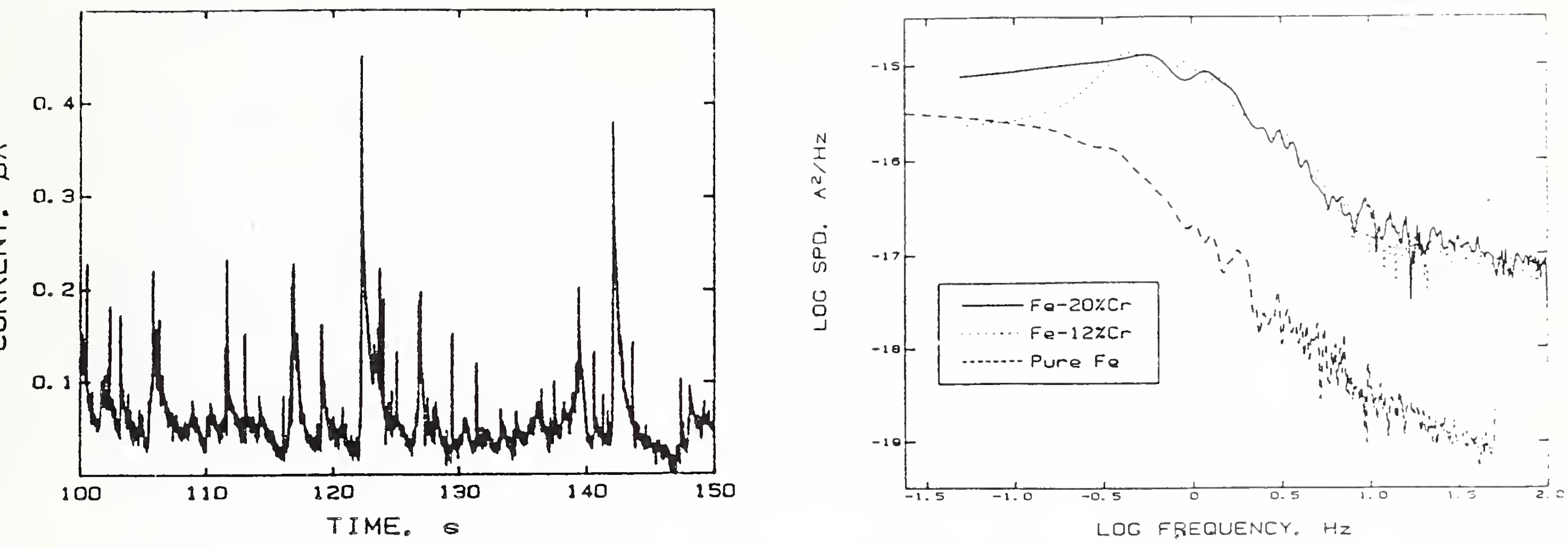


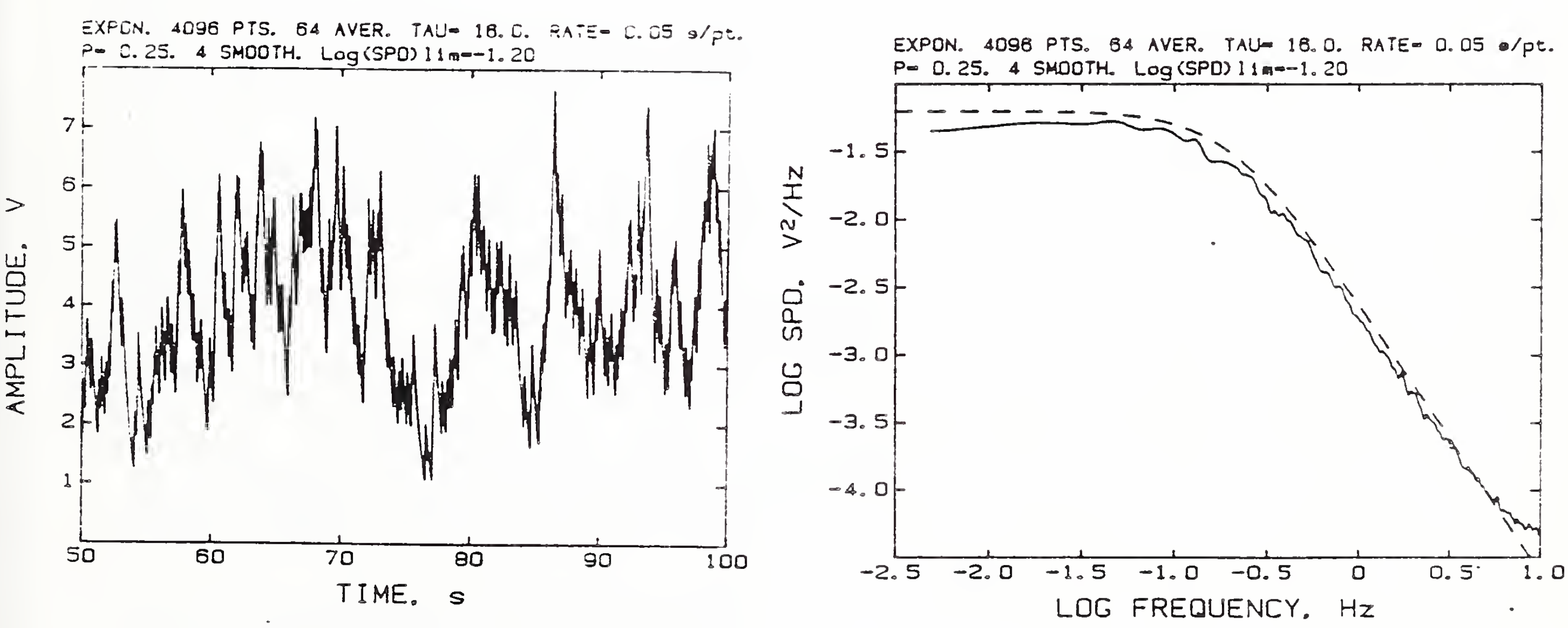
Figure 2. In oxygen-containing 0.5M NaCl electrolyte, an anodic current associated with pitting begins to flow at about 0V/SCE on both Ni and Ni_3Al. The alloy does not show current corresponding to pitting of Al.



A

B

Figure 3. Current fluctuations recorded before pit initiation on various materials. A) Part of time record, taken on pure Fe in borate buffer + NaCl. B) Power spectra.



A

B

Figure 4. Computer simulation of random transients. Exponential decay. A) Part of the time record. B) PSD of the transients after ensemble averaging. Dashed curve is spectrum calculated from model.

ELECTRODEPOSITION

David S. Lashmore

The Electrodeposition Group is responsible for measurements and standards associated with electrodeposited metals and alloys. The objectives of the group are: (1) the determination of the critical mechanistic, materials, and process variables controlling the structure/property relationships of electrodeposited coatings and the development of approaches that will result in entirely new materials; (2) the provision of standards such as coatings thickness standards, dye penetran crack standards, and tin-lead alloy standards; (3) the development of new standards requiring electrodeposition for their fabrication or utilizing the unique properties of electrodeposited alloys; (4) the provision of government expertise to industry, through research associates and standards organizations, and to the government agencies, through appropriate contracts and consulting arrangements.

Following are some of the many areas in which coatings are important to the commerce of the United States: (1) Strategic Materials - it has been shown that appropriate alloy coatings can provide a 30% savings of imported raw chromium. Research on new alloys in progress indicated that for many applications, coatings can replace bulk stainless steels. (2) Corrosion-electrodeposited coatings play an important role in corrosion protection. It has been estimated that the cost of corrosion to the U.S. economy is in excess of 200 billion dollars per year. (3) Wear - The cost of wear to the U. S. economy has been estimated at about 50 billion dollars per year. Electrodeposited coatings play an important role in improving wear properties and surface coatings can be optimized for particular wear situations. (4) Electrodeposition Industry - specifically plays an essential role in the United States economy. For example, almost 900K tons of electrogalvanized sheet and strip are produced annually with an estimated impact of about $150M per year projected by 1990, approximately 550K tons of metal coated wire and wire products are produced per year, tin plate accounts for 8800 tons with a dollar impact of $120M, and foil production accounts for about $200M per year. (5) Magnetic Materials - Most hard disk drives are produced utilizing electrodeposition technology which thus has an important impact on the United States computer industry. (6) Processes - including decorative coatings, electroforming (compact discs), and electronic application (contacts, PC boards etc.) are so important that without electrodeposited coatings much of our current industry would not be able to function in its present form. (7) Advanced Alloys - new approaches using electrochemical deposition have resulted in new materials whose properties can be tailored on a near atomic scale. Basic work is now going on in electroformed intermetallics, advanced composites, and new magnetic materials.

FY 88 Significant Accomplishments

- Production of Coating Thickness Standards was increased by 15% over previous years to 7,000 standards. This was accomplished in part by converting to a microcomputer controlled measuring system.

- Developed three new coating thickness standards as requested by the American Welding Institute. These coating thickness standards provide standardization values for designating the delta ferrite content of

austenitic stainless steel weld metal formed when the weld metal solidifies.

- Abrasive wear rate was reduced by 50% by incorporating B_4C (tetra boron carbide) into a chromium matrix, compared with hard chromium. An alternate system showing promising results is a particulate composite alloy using a nickel matrix in which SiC is incorporated. Preliminary tests show a reduction in wear of 44% compared with hard chrome. (note that hexavalent chromium is highly toxic)

- Problems of interfacial diffusion in metal matrix composites are being addressed by electrodeposition of diffusion barriers in a joint program between the National Institute of Standards and Technology (NIST) and American Cyanamid Corporation. Barriers for carbon/nickel interdiffusion have been electrochemically produced on graphite fibers. Alloys of cobalt with tungsten have shown significant benefit in suppressing interdiffusion.

- The mathematical model of alloy deposition onto moving filaments considering potential distribution in solution and within the working electrode, has been expanded to include the concentration polarization. A bench top cell has been completed and successfully operated.

- Recent developments in the Electrodeposition Group on modulating the microstructure of chromium are expected to lead to significant advances in wear performance for many applications. Microlayered chromium has been found to have apparent anisotropic deformation which depends on layer spacing. Microhardness of microlayered chromium was found to be 1500-1800 KHN/25gf at layer spacings of 1 to 3 nm.

- The composition and structure of aluminum alloys electrodeposited from chloroaluminate electrolytes can be rigorously controlled by melt composition, temperature (120-500 °C), and electrode potential. In the Al-Mn system, the direct deposition of amorphous, metastable (icosahedral and decagonal), and crystalline (Al_6Mn + unknown) phases has been demonstrated. This is the first evidence for the direct electrodeposition of aluminum intermetallic compounds.

- Al-Ti alloys containing up to 40 wt % Ti have been electrodeposited from chloroaluminate electrolytes at 150 °C. Heat treatment converts the deposit to a mixture of Al and Al_3Ti as predicted by the equilibrium phase diagram. The direct deposition of AlTi and Ti_3Al should be possible from this electrolyte at elevated temperatures. Forming intermetallic compounds by electrodeposition may overcome fabrication and machining problems inherent to the material and which presently limit other processing techniques.

- Tin/Lead SRMs certified for specific assigned mass per unit area and specific composition are being developed for sale by the Office of Standard Reference Materials. This alloy (60Sn-40Pb) is in wide use by the electronics industry and the production of SnPb SRM's has been requested by the American Society of Testing and Materials.

- Significant improvements in production techniques for electrochemically producing artificial superlattices have been made. The new technology allows compensation for random disturbances in the electrolyte and has yielded samples significantly better than those produced by other processing technologies. Magnetic studies of these new atomically controlled superlattices have yielded new data on the temperature dependence of the saturation magnetization, have proven the absence of a magnetic dead layer at the interface, and are expected to provide other new insight into metal physics.

Metal Matrix Composites
C. R. Beauchamp, S. A. Claggett, C. E. Johnson, D. R. Kelley, D. S. Lashmore, J. L. Mullen, P. N. Sharpless, G. R. Stafford, and N. S. Wheeler

Electroforming Metal Matrix Composite Precursors - A two year investigation of interface behavior in metal-matrix composites has been initiated in co-operation with the American Cyanamid Corporation and in collaboration with members of the Metallurgy Division and the Center for Analytical Chemistry. A literature study revealed that much of the research on metal-matrix composite interfaces has been done by the Soviets, and many of the Russian papers have been translated (K. W. Pratt). The focus of this year's research has been the stabilization of the interface between nickel and graphite so that deterioration of the mechanical properties of graphite might be reduced or eliminated at elevated temperatures. The problem is the interdiffusion of nickel and carbon at high temperatures (i.e., greater than 600 °C), which results in a brittleness and decrease in diameter of the fiber. The research effort has focused on the electrodeposition of an alloy containing a carbide-forming element directly on the graphite fiber prior to the outer nickel layer. Of the alloys considered, Co-W has been successfully electrodeposited from aqueous solution onto graphite fibers of polyacrylonitrile (PAN) base. Scanning Electron Microscopy (SEM) (A. J. Shapiro) has indicated that for the Co-W coated fibers in a nickel matrix: 1) no change in shape or measurable decrease in the size of the fibers occurs as a result of annealing at 800 °C for 24 hours, and 2) nickel diffused through the Co-W layer but had not penetrated the fiber (see figure 1). This dramatic improvement in the tolerance of the graphite fibers for nickel is apparently due to a very thin layer of an unidentified species or combination of species. A Transmission Electron Microscopy/Electron Diffraction (TEM/ED) study has just been initiated to characterize the interface, preliminary X-ray diffraction studies of annealed Co-W coated fibers have shown Co_3W_3C and Co_6W_6C to be present. A comparison will be made between the phases found by TEM/ED and those predicted by the quaternary phase diagram for C-Co-Ni-W. Mechanical property studies by fiber punch-out and tensile testing are also underway.

Mathematical Modeling - The efforts in this past year have been concentrated on the completion of the mathematical modeling of the deposition process onto moving wire. Previous assumptions with respect to the linear dependence of the resistivity of the alloy have been eliminated and the correct dependence based on experimental data has been included in the form of a fitted polynomial over published values for this property. In addition, the finite difference algorithm used to solve the partial differential equations has been upgraded to include a treatment of unequal intervals during the

determination of the grid spacing. This allows a greater resolution on regions of higher gradients for the parameter under consideration and a greater stability during the convergence of the method. The two most important parameters while solving the coupled differential equations are the concentration of the active species and the potential distribution in solution. Considerable effort has been given to the choosing of the proper transformation function while solving for these parameters. Unfortunately the same transformation for the radial distance is not suitable for the whole treatment because the potential distributions behave logarithmically in the neighborhood of the electrode surface while the concentration profile behaves logarithmic at the surface, but decays to the negative power of one third at the jet exit. The best transformations found for the potential and concentration profiles are represented in figure 2.

Deposition of the Aluminum Alloys - the Matrix - Aluminum alloys can be electrodeposited from molten salt electrolytes containing $AlCl_3$, NaCl and the chloride salt of the solute metal at temperatures as low as 110 °C. For example, the addition of small quantities of $MnCl_2$ to the chloroaluminate melt results in the deposition of a wide variety of binary aluminum-manganese alloys. The electrodeposition of these alloys from molten salt electrolytes offers many advantages over conventional solidification techniques. Not only can one rigorously control the alloy composition (through melt composition and electrode potential), but the span of operating temperatures possible (120-500 °C) allows for the deposition of a variety of structures. By altering the temperature of the electrolyte during deposition, one can electrodeposit homogeneous films of amorphous, metastable, and equilibrium phases. The ability to electrodeposit thick, uniform films of the icosahedral and decagonal phases for instance, may lead to the first mechanical property measurements of these very important materials.

Similarly, Al-Ti alloys can be electrodeposited from chloroaluminate electrolyte containing Ti^{+2}. The deposition potential of titanium is very close to that of aluminum; consequently, a broad range of alloy compositions is possible as long as the concentration of Ti^{+2} is sufficient to support the partial current required for titanium deposition. Presently, the electrolytes contain about 40 mM Ti^{+2}. Electrodeposition from this electrolyte at 150 °C and at moderate current densities (1-20 mA/cm^2) results in alloys containing 10 -20 wt.% Ti which convert to a mixture of Al and Al_3Ti upon heat treating at 600 °C. At very low current densities (100 μA/cm^2), the titanium current efficiency increases significantly and we have observed structural evidence (x-ray diffraction) for the direct electrodeposition of AlTi. Forming intermetallic compounds by electrodeposition, particularly the titanium-aluminides, may help overcome fabrication and machining problems inherent to the material and presently limiting other processing techniques.

Direct Adhesion Testing of Cobalt-Tungsten Alloys on Fibers - Techniques were developed to measure adhesion of coatings to fibers as small as 7μm. The technique incorporates a dynamic microhardness instrument (U. S. Patent 4,699,000) using a custom made diamond indentor. These methods revealed that alloys of Co-W adhere significantly better to silicon carbide than copper or nickel. Tests are currently underway on graphite and on other coating

alloys.

Composition Modulated Deposits for Application as Neutron Mirrors
D. S. Lashmore, C. E. Johnson, and U. Bertocci

Research on electrodeposited composition modulated coatings for possible application as neutron mirrors resulted in the development of an electrolyte to produce nickel-manganese and nickel-tin microlayered deposits. Galvanostatic pulse plating techniques were developed to deposit discreet alternating layers of nickel/nickel-manganese and nickel/nickel-tin from the single electrolyte. A scanning electron micrograph of a representative nickel/nickel-manganese layered deposit is shown in figure 3 with the associated energy dispersive x-ray analysis of the composition of the coating cross-section.

Wear Resistant Coatings for the Printing Industry
C. E. Johnson, D. R. Kelley, J. L. Mullen, and D. S. Lashmore

A one year study of the wear of electrodeposited coatings on wiper blades used on water-wipe currency printing presses at the Bureau of Engraving and Printing was completed. The purpose of the study was twofold, to extend the life of the wiper blades beyond that obtained with unplated steel blades and to compare the results of laboratory accelerated wear testing with in-situ wear testing of coated blades on the printing presses. Four types of chromium coatings (BEP hard chromium, milky non-cracked chromium, direct current deposited duplex, and pulse plated duplex) were deposited on W-1 wiper blades and evaluated for wear performance. The coatings were obtained by direct current (DC) plating, galvanostatic pulse plating, and by varying operating parameter, such as temperature and current density. The laboratory and in-situ wear tests ~~results~~ of the four types of chromium coatings are compared in figure 4. It was found that coating W-1 blades with 100 μm of BEP hard chromium, the life of the blades could be extended 10-15 times the life of the uncoated blades. The laboratory accelerated wear tests of the pulse plated duplex chromium coatings show a 50% improvement in wear performance over the BEP hard chromium; whereas, the in-situ wear tests show a 30% improvement. The trends indicated by the in-situ testing provided a good check of the laboratory testing.

Three wiper blades (W-1, W-2, and W-3) are incorporated in each water-wipe press. The wear environment of the three blades range from wet abrasive to dry abrasive. The wet abrasive environment has been investigated with the coatings on W-1 blades. W-3 blades are subjected to the dry abrasive environment. In-situ wear results of hard chromium coated W-3 blades indicated that the life was extended 4-6 times over the unplated blades, but this was only one-half the life extension noted for W-1 blades. This result is indicative of the severity of the dry abrasive environment. Another type of coating, particulate composites, were investigated for wear performance in the laboratory to address the severity of the dry abrasive environment of the W-3 blades. The composites were either a nickel or chromium matrix with silicon carbide or boron carbide particles. A summary of the wear rates of the composites are compared to the steel and hard chromium coated blade sections in figure 5. Composites of 5 μm boron carbide particles in either

the nickel or chromium matrix wore up to 2.5 times better than the hard chromium.

An extension of the work on pulse plated duplex chromium deposits has led to an investigation into the deposition of structure modulated (SM) chromium coatings. Modulated deposits were prepared with layer spacings from 1-500 nm. X-ray diffraction of these deposits have indicated that different structures (BCC, HCP, and FCC) can be obtained but maintaining these as stable structures has not been achieved except for the normal BCC structure. The layers that have been induced, figure 6, appear to be caused by texturing (preferred orientation) of which control has been achieved by controlling the pulsing parameters, current density, and electrolyte temperature. Microhardness testing of these deposits resulted in hardness values of 1500-1800 knoop hardness numbers when measured at loads of 0.098 newtons (10 gf) and 0.245 newtons (25 gf). These hardness values compare to 1000 knoop numbers for hard chromium.

Standard Reference Materials
H. J. Brown, H. G. Brown, D. R. Kelley, D. S. Lashmore, and P. N. Sharpless

This year, the Electrodeposition SRM production staff certified over 7,000 coating thickness standards, a 15% increase over FY87. Included in this years production, were three new coating thickness standards which were requested by the American Welding Institute and were completed six months ahead of schedule.

The increase in production can be attributed to the conversion of the S-100 computer system to the new micro computers, and modified programs.

Research on the Standard Reference Material, Sn-Pb is rapidly concluding and production is slated to begin by November 1, of this year. These standards will be certified for composition and thickness (specific mass per unit area).

Work is in progress on a new standard reference material, Ni-P. This standard will be certified for thickness (specific mass per unit area and phosphorous content. A second x-ray florescence unit has been purchased which will be dedicated solely to detecting light elements, and will be used to certify the Ni-P standard for thickness and phosphorous content.

Planning has begun to redesign the award winning, Scanning Electron Microscope Certification Standard. It is proposed to change from the present Ni-Au layered structure to possibly a Ni-Cu or Ni-Ag system. The Electrodeposition Group will contract to develop this new plating system only. Certification of the standard will be done elsewhere at NIST.

Electrochemical Deposition of Artificial Superlattices
D. S. Lashmore, R. R. Oberle, S. A. Claggett

Significant progress has been made in the deposition and characterization of Copper-Nickel composition modulated alloys under sponsorship of the IBM Corporation. A refinement of a potentiostatic deposition system has been made that makes use of a high speed coulometer and a mini computer as a

feedback and control mechanism. This system allows for control of deposition potential while monitoring the charge used in the plating process. Application of this system to copper-nickel allows for the characterization of the structure and perfection of superlattices as a function of these plating parameters. X-ray diffraction data in figure 7 shows the development of X-ray satellites as a function of the nickel reduction potential. Alloys produced with the new feedback and control system have demonstrated enhanced saturation magnetization; Ms, from room temperature to 4 °K. The value of Ms for the electrochemically produced alloys is higher than measured for any Cu-Ni superlattice produced by the competing technologies of sputtering or molecular beam epitaxy. This data has also demonstrated that contrary to current theory there is no magnetic dead layer at the copper-nickel interfaces. Finally, a new magnetic aftereffect has been observed that continues for some tens of seconds.

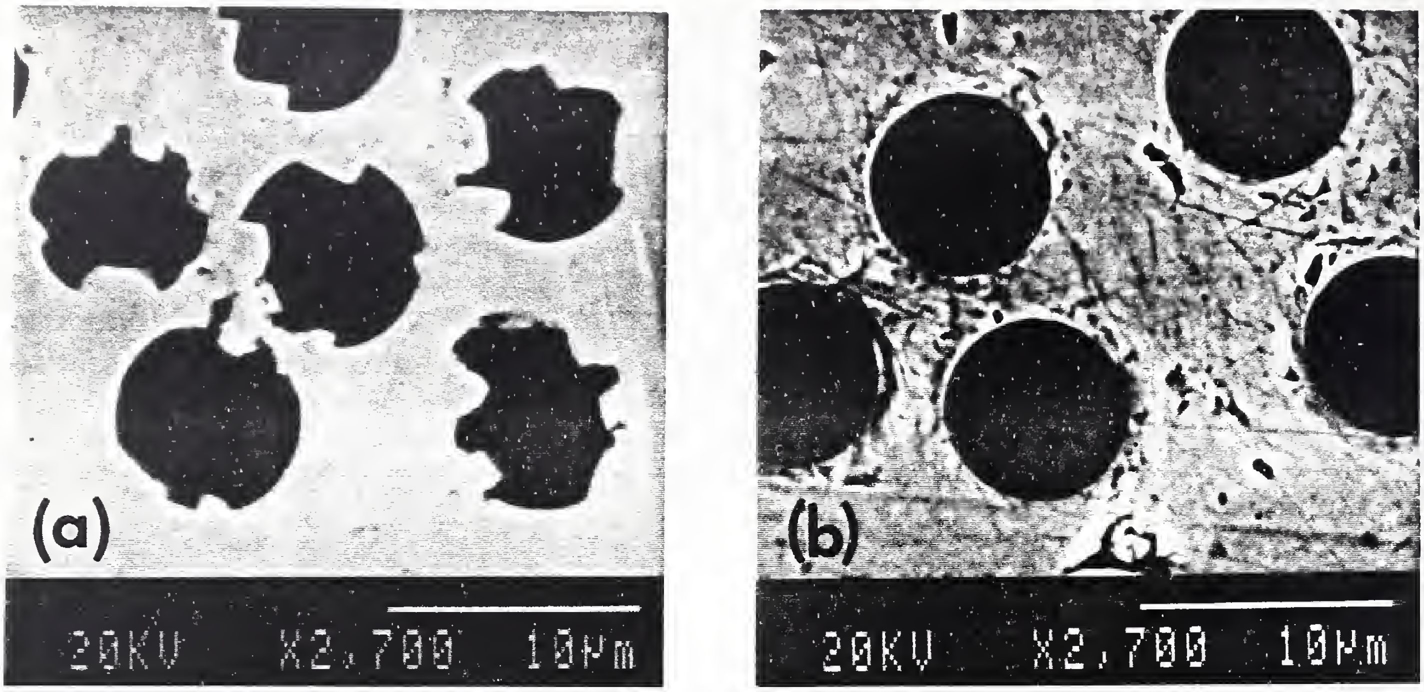


Figure 1. Comparison of the behavior of uncoated graphite fibers (a) with Co-W coated fibers (b) in a nickel matrix heated to 800 degrees centigrade.

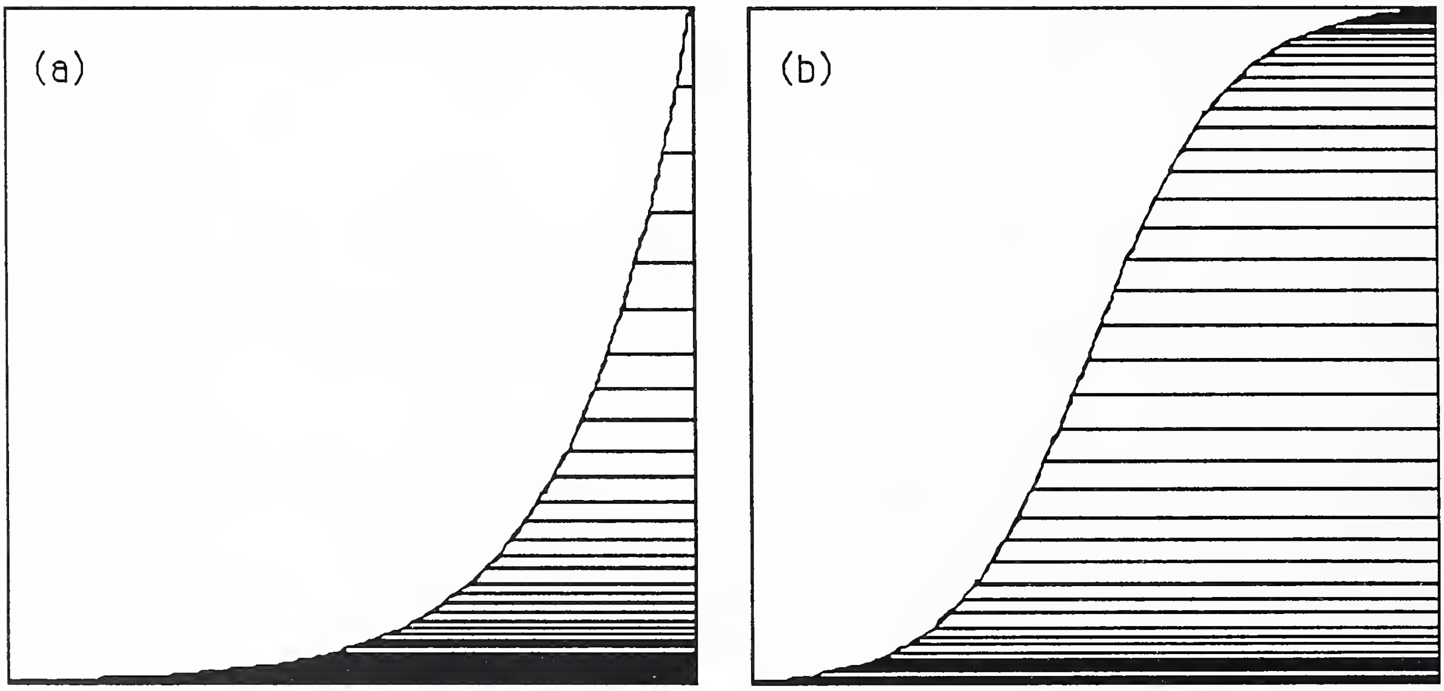


Figure 2. Transformation functions of potential (a) and concentration (b) profiles used to improve the finite difference model convergence.

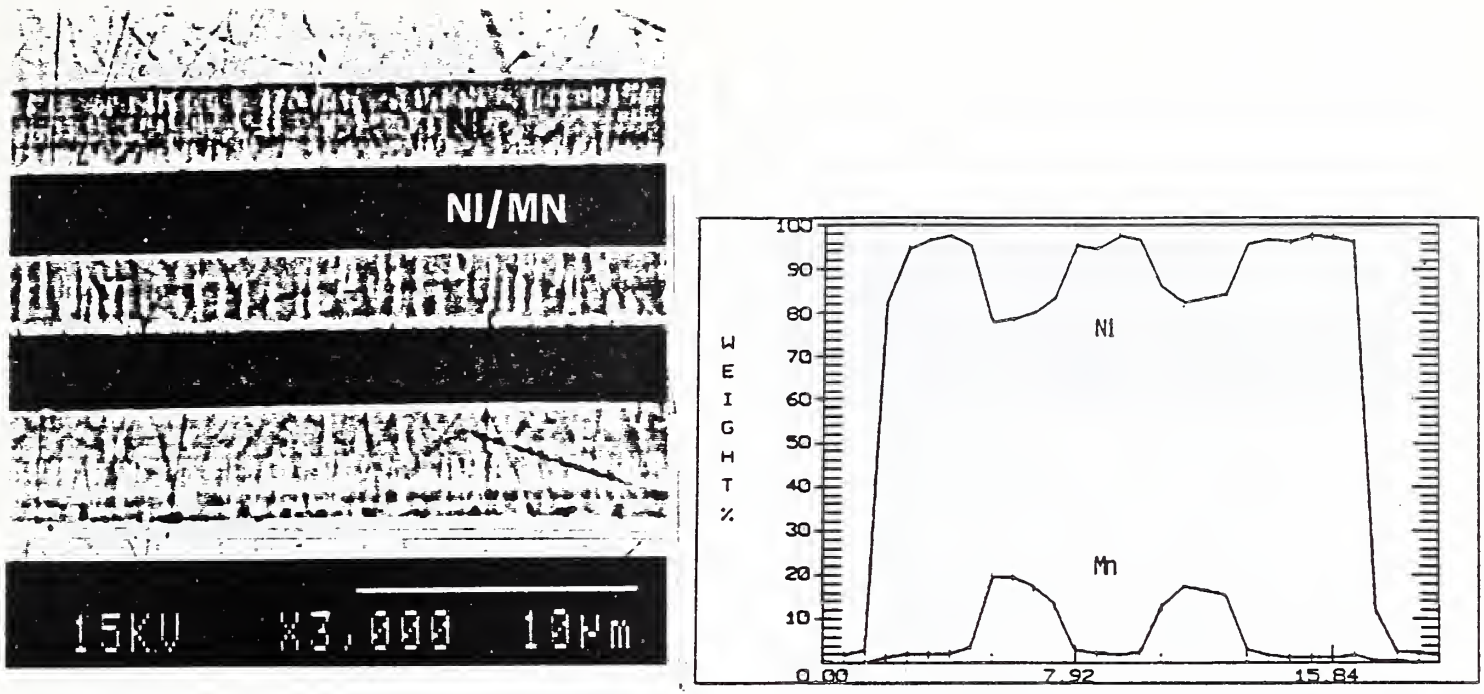


Figure 3. An SEM micrograph and an EDAX trace of a sectioned Ni-Mn compositionally modulated alloy coating under development for neutron mirrors.

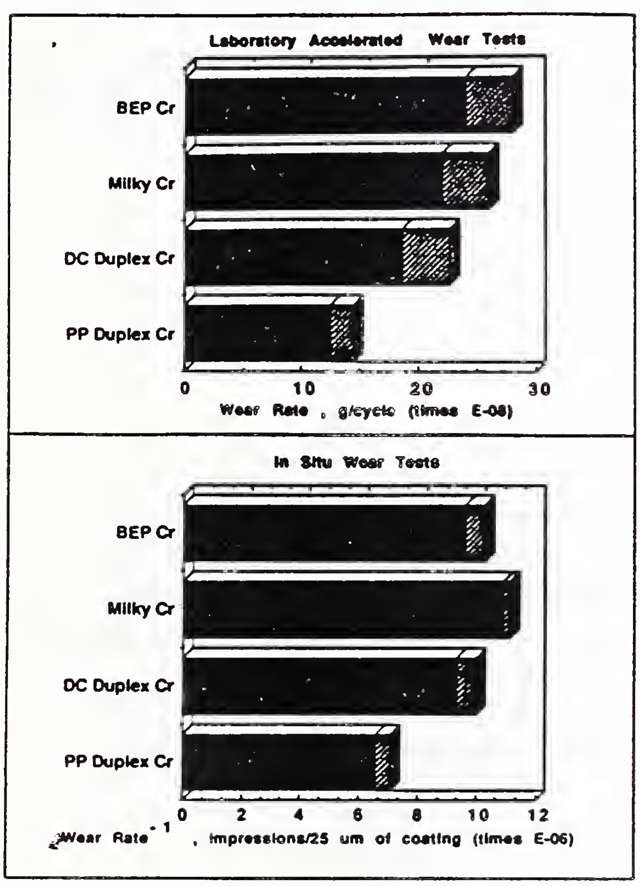


Figure 4. Laboratory and Insitu wear test comparison for experimental high performance wear resistant chromium electrodeposits.

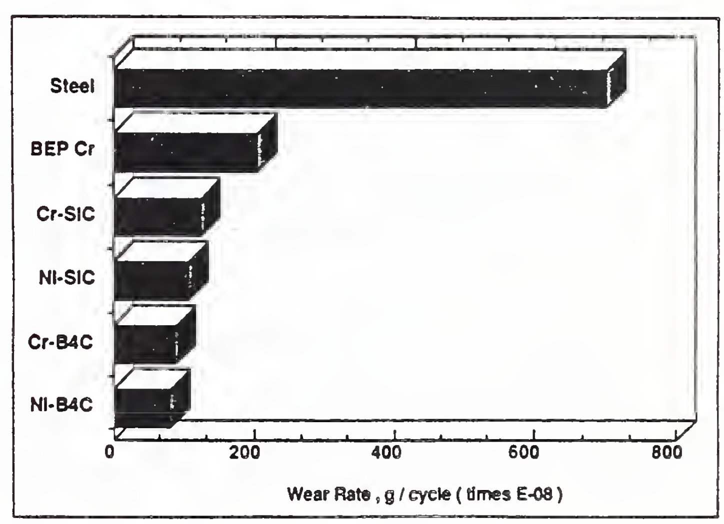


Figure 5. Comparison between electrochemically produced particulate composites with hardened steel and chromium coated steel controls.

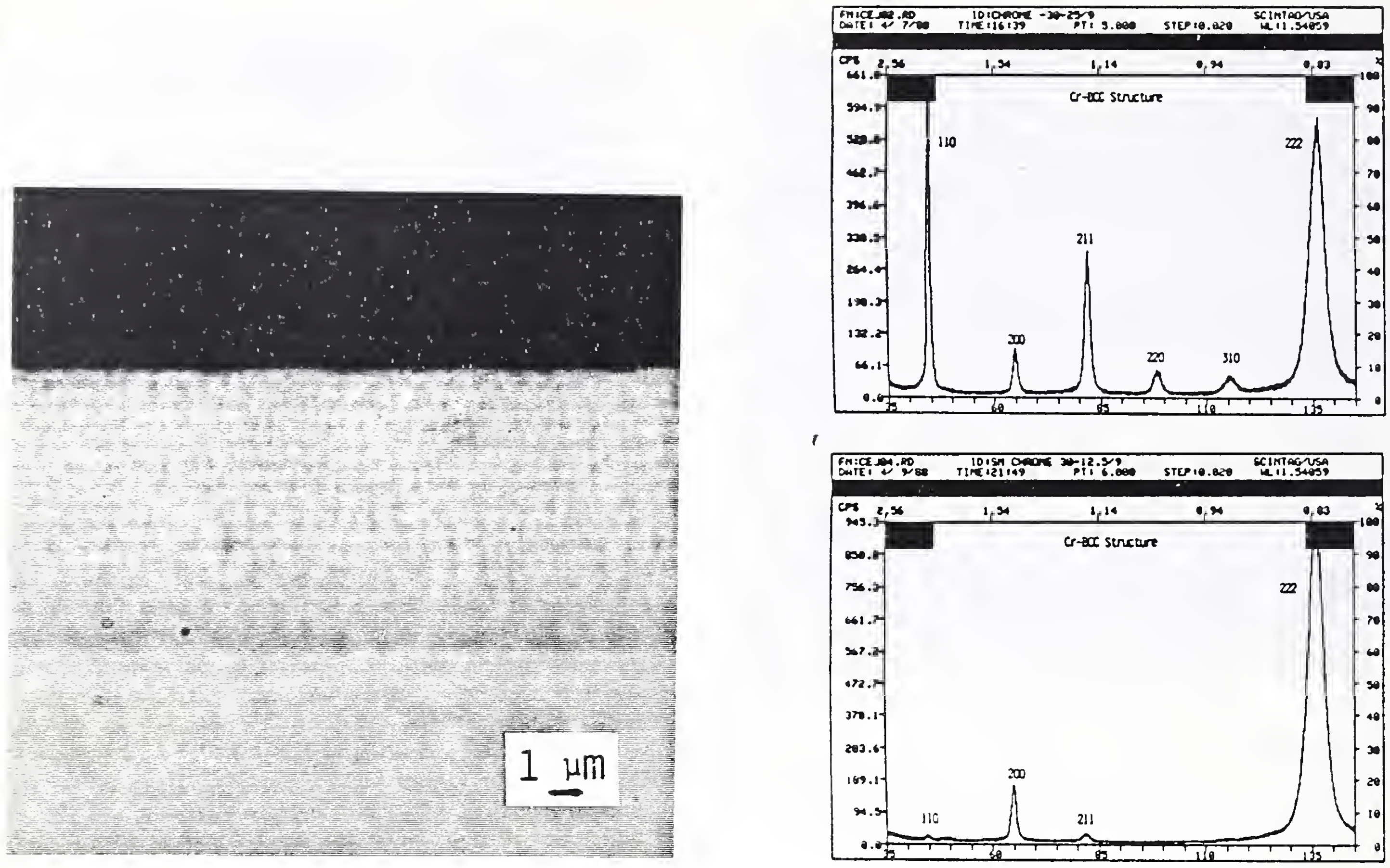


Figure 6. Structure modulated chromium alloys. The layers are the result of texturing (preferred orientation) of the chromium BCC structure controlled by the plating parameters.

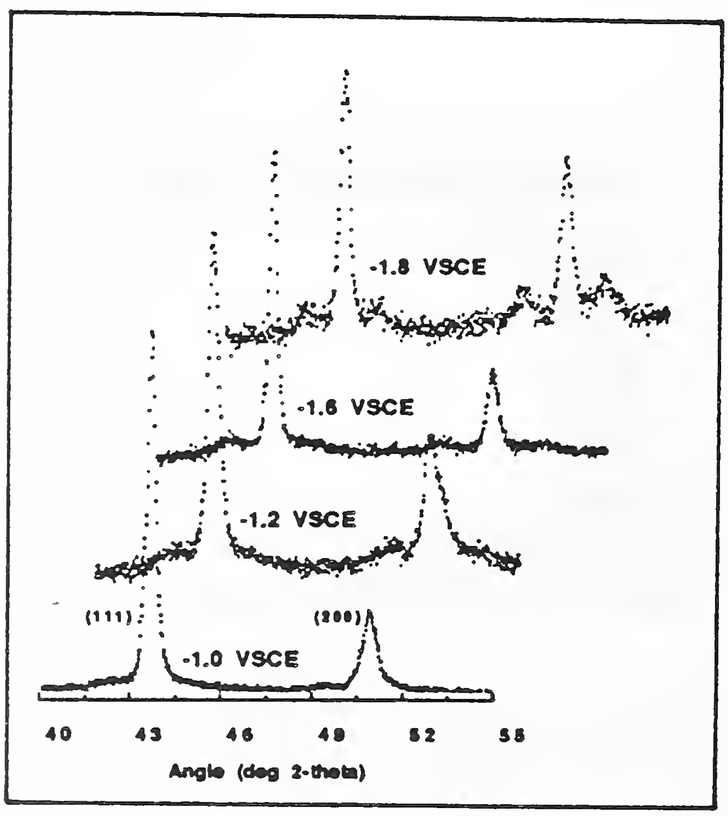


Figure 7. An X-ray diffraction pattern of electrochemically produced Cu-Ni superlattices showing how the perfection (satellite intensity) increases with nickel potential.

MAGNETIC MATERIALS

Lawrence H. Bennett

The Magnetic Materials Group is concerned with the measurement of the magnetic properties of advanced magnetic and superconducting materials. The objectives are (1) to relate the metallurgical, magnetic and electronic structure to the magnetic and superconducting properties of materials, (2) to develop new and improved magnetic measurement methods, (3) to develop magnetic reference standards, (4) to apply magnetic phenomena to the nondestructive evaluation of materials and structures, and (5) to provide expertise to industry, universities and other government agencies.

Magnetic materials are important to the commerce of the United States. For example, the sales of soft magnetic materials (primarily for information and data storage) amounts to more than 25 billion dollars per year. The sales of hard magnetic materials (primarily for motors) are more than one billion dollars per year and have been recently increasing due to the discovery of supermagnetic rare-earth alloys. Magnetic methods of nondestructive evaluation are used for quality control practically everywhere steel is used. The new high-temperature superconductors have important potential applications, both near-term and future.

FY 88 SIGNIFICANT ACCOMPLISHMENTS

- Transient effects in the magnetic hysteresis loops of the high T_c superconductor $YBa_2Cu_3O_{7-x}$ were measured at a variety of temperatures. A dynamic magnetic viscosity ("flux flow"), with a decay time of ~10s, as well as Anderson-Kim flux creep at larger times, were both observed in the same sample.

- The local topology of the atoms in the high T_c superconductors was investigated using a modified Wigner-Seitz construction and shown to exhibit three-dimensional chemical bonding. The La-Cu-O type (≈40K) and the Y-Ba-Cu-O type (≈90K) superconductors both were found to display the same bonding characteristics.

- Thin films of the high T_C superconductor, Bi-Sr-Ca-Cu-O, were deposited by laser ablation onto a ZrO_2 substrate. By using the magnetically-modulated-microwave-absorption (MAMMA) technique, we have been able to show how the film quality is affected by the substrate temperature and the post anneal. These quality differences are obscured in dc resistivity measurements.

- Mössbauer spectra, as a function of oxygen concentration, of the high T_C superconductor $RBa_2Cu_3O_{7-x}$ [R=Y, Pr, Er] were obtained for samples having 3% Fe substituted for Cu. The results established that the iron acted as an excellent probe for the anti-ferromagnetism associated with the oxygen-deficient samples.

- Magnetic measurements on Ni/Cu compositionally-modulated thin films produced by the Electrodeposition Group with their improved electroplating procedure exhibited (1) higher magnetizations and (2) smaller temperature dependences of the magnetization than any reported previously by any technique. These results demonstrate that electrodepositing nanometer thick layers of the highest quality is feasible.

- An experimental system for detailed analysis of the magnetic Barkhausen noise in commercial steels has been designed and constructed. This system, which measures the (1) noise pulse amplitude distribution profile, (2) power spectral density, (3) pulse number spectrum and (4) total noise power, will provide a scientific basis for the use of Barkhausen noise as a nondestructive evaluation tool.

- A series of $Ag+Fe_3O_4$ thin films prepared by R.F. diode sputtering of a compositionally split target to intentionally create a composition gradient in the series of deposits revealed a grain size transition at ≈30 wt. % Ag to a nanometer size granular morphology. These nanocomposites were characterized by Mössbauer spectroscopy, x-ray diffraction and, of especial importance, field-emission scanning-electron microscopy (FESEM).

Compositionally-Modulated-Alloy Thin Films
L. H. Bennett and L. J. Swartzendruber

The Electrodeposition Group has established that it is possible to produce compositionally-modulated alloys (CMA) with individual layers in the nanometer range by electrochemical deposition using with an improved electroplating procedure. A series of magnetic measurements on Ni/Cu compositionally-modulated thin films produced with this improved electrodeposition was carried out. The hysteresis loop measured with the field perpendicular to the plane of the foil showed a coercive force less than the parallel loop. These foils also exhibited (1) higher magnetizations and (2) smaller temperature dependences of the magnetization than any reported previously by any technique. The temperature dependences of the magnetization tracked the x-ray satellite intensities, as a function of Ni overpotential (figure 1). These results demonstrate that electrodepositing nanometer thick layers of the highest quality is feasible and that the magnetic measurements constitute a meaningful characterization tool for them.

High-Temperature Superconductors
L. J. Swartzendruber, L. H. Bennett, and R. D. Shull

Among the most technologically significant properties of the new high-T_c superconductors are magnetic flux pinning and the associated critical currents including their dynamic characteristics. Transient effects in the magnetic hysteresis loops of the high T_c superconductor $YBa_2Cu_3O_{7-x}$ were measured at a variety of temperatures. A dynamic magnetic viscosity ("flux flow"), with a decay time of ~10s, as well as Anderson-Kim flux creep at larger times, were both observed in the same sample for the first time. A major question always is to what extent are these properties intrinsic to the material and to what extent are they due to the grain boundaries and other microstructural characteristics. Since high quality single crystals can only be produced in very small sizes, very sensitive magnetic measurements are required. As illustrated in figure 2, this capability is now present in the Metallurgy Division. This figure indicates transient effects in the pinched central region of the loop. An important new flux "depinning" phenomenon has also been observed in high-field hysteresis loops in several samples.

The local topology of the atoms in the high T_c superconductors was

investigated using a modified Wigner-Seitz construction and shown to exhibit three-dimensional chemical bonding. The La-Cu-O type (≈40K) and Y-Ba-Cu-O type (≈90K) superconductors both were found to display the same bonding characteristics. An example of the bonding in two extremes is shown in figure 3.

Continuing a cooperative program with the Johns Hopkins University Applied Physics Laboratory, thin films of the high T_c superconductor, Bi-Sr-Ca-Cu-O, were deposited by laser ablation onto a ZrO_2 substrate. By using the magnetically-modulated-microwave-absorption (MAMMA) technique, we have been able to show how the film quality is affected by the substrate temperature and the post anneal. These quality differences are obscured in dc resistivity measurements.

Mössbauer spectra, as a function of oxygen concentration, of the high T_c superconductor $RBa_2Cu_3O_{7-x}$ [R=Y, Pr, Er] were obtained for samples having 3% Fe substituted for Cu (e.g., see figure 4). The results established that the iron acted as an excellent probe for the anti-ferromagnetism associated with the oxygen-deficient samples. In general, the occurrence of a high T_c was associated with the disappearance of the antiferromagnetism.

Nanocomposite Materials

R. D. Shull, L. J. Swartzendruber, and L. H. Bennett

Composite materials (immiscible metals and oxides with a size scale of the order of nanometers) have been shown to possess unusual composition-dependent electronic and magnetic properties. These materials provide a unique laboratory for the atomic engineering of specific material properties.

Thin films of immiscible Ag and Fe_3O_4 were prepared with varying compositions by cosputtering (R.F. diode) the two constituents from a compositionally-split target. A transition to a morphology of nanometer-sized regions of the two constituents was observed at a composition of ≈30 wt. % Ag (see figure 5). This micrograph is the first view of the surface of a nanocomposite material by means of a field emission scanning electron microscope (FESEM). [This microscope possesses the very high magnification of the transmission electron microscope (TEM), but does not require the special specimen preparation techniques.] Coincident with the development of nanometer-sized grains, the magnetic behavior of the thin films also changed. Ferrimagnetism was observed by Mossbauer spectroscopy for smaller Ag contents, while superparamagnetic behavior was exhibited by those thin films containing ≥ 30 wt.% Ag. Nanocomposites of $Ag+Fe_3O_4$ were also prepared by cosputtering from mechanically mixed powder targets. However, in this latter case the critical composition required for the development of superparamagnetic behavior was only 5 wt. % Ag. The difference in critical compositions in the latter case is ascribed to the much greater interaction between the species in the sputtering plasma. These nanocomposite materials have potential for vertical recording and in magneto-optical information storage.

Another way to prepare nanocomposites, the sol-gel technique, is under investigation in cooperation with Dr. J. Ritter of the Ceramics Division. Preliminary x-ray diffraction and Mössbauer data appear encouraging.

Magnetic NDE

L. J. Swartzendruber and H. Ettedgui*

*Guest Scientist, Nuclear Reasearch Center - Negev, Israel

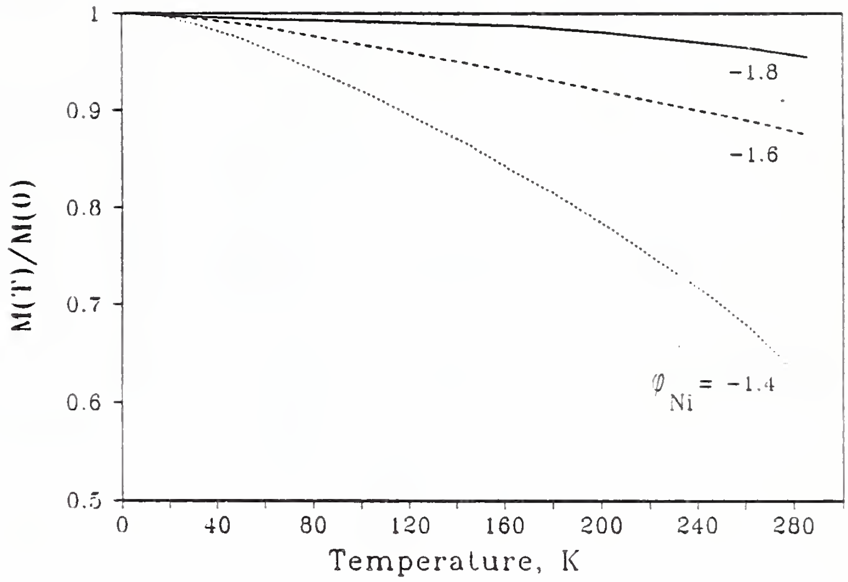
Magnetic particle inspection, magnetic flux leakage testing, and magnetic permeability measurement, are nondestructive evaluation techniques widely used in industry, the first two for defect detection, and latter for property determinations such as ferrite content in stainless steel welds. A number of methods (Barkhausen noise, magnetoacoustic emission, magnetomechanical damping, and ac magnetometry) have been used on a limited scale but require further development to increase their reliability and make them more widely applicable. Finally, some magnetic property measurements (e.g. saturation magnetization, coercivity, and initial permeability) contain a wealth of information concerning material properties that has barely begun to be exploited. Current effort on these techniques in the Metallurgy Division include magnetic particle inspection standards and Barkhausen noise analysis.

A large number of workers have shown that measured properties of the Barkhausen noise can be related to material properties such as hardness, grain size, internal stress, and defect density. When usable, the Barkhausen methods is very attractive because high production rates are possible. However, the relationship between the observed noise and a material property is always heuristic and is generally poorly understood from a fundamental point of view which could lead to erroneous conclusions. There are many ways to characterize Barkhausen noise, including (1) the noise pulse amplitude distribution profile, (2) the power spectral density, (3) the pulse number spectrum, and (4) the total noise power. Currently available equipment uses one or another of these methods to produce a "Barkhausen number" which changes when a material property changes, but the same method does not appear to be used by any two pieces of equipment. Our purpose has been to develop an experimental arrangement which will have the flexibility to calculate these "numbers" from the characteristics of the observed noise as well as to allow a detailed analysis of the noise from a fundamental point of view. This will be accomplished primarily by software analysis of digitized noise signals. An example of a Barkhausen noise spectrum and a power density spectrum of this noise obtained using digital techniques is shown in figure 6. A theoretical model of an elementary Barkhausen jump is being examined, using numerical computer procedures, for evidence of deterministic chaos. Effort during the coming year will focus on further development of our Barkhausen system and associated software and on obtaining and analyzing Barkhausen noise from selected steels with a variety of microstructures. The goal is to develop materials with very well characterized noise properties that can be used as standards to compare the output from different types of noise analyzers.

During the current fiscal year a second version of the military standard for magnetic particle inspection (MIL-STD-1949) was prepared and coordinated (work supported by the U.S. Army Materials Technology Laboratory). Valuable comments were obtained from over fifty companies. We also are participating in conversion of this standard to an ASTM document.

Fig. 1.

Saturation magnetization, M(T), normalized to the saturation magnetization at T=0, versus temperature for three different Ni/Cu multilayer films. All three were produced by electrodeposition with identical electrochemical parameters to produce 440 layers of ~30ÅNi/~30ÅCu, except for changing the Ni overpotential, as shown.



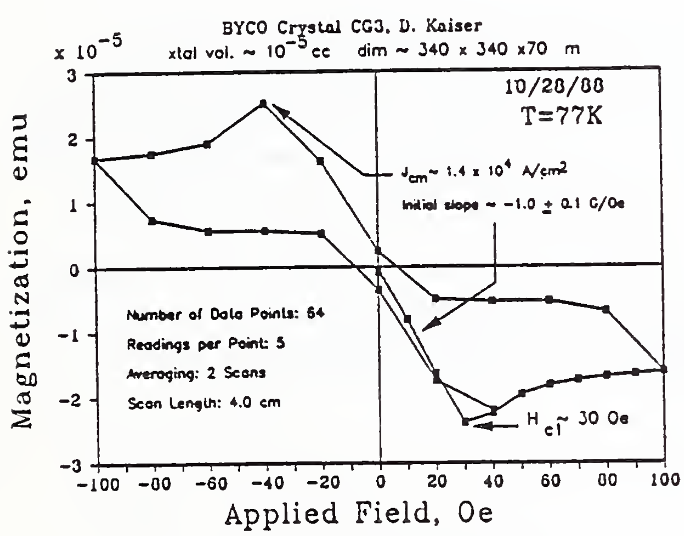


Fig. 2.

Low-field hysteresis loop in a high-quality $YBa_2Cu_3O_{7-x}$ single crystal weighing about 60 μg, measured in the Metallurgy Division's new SQUID magnetometer.

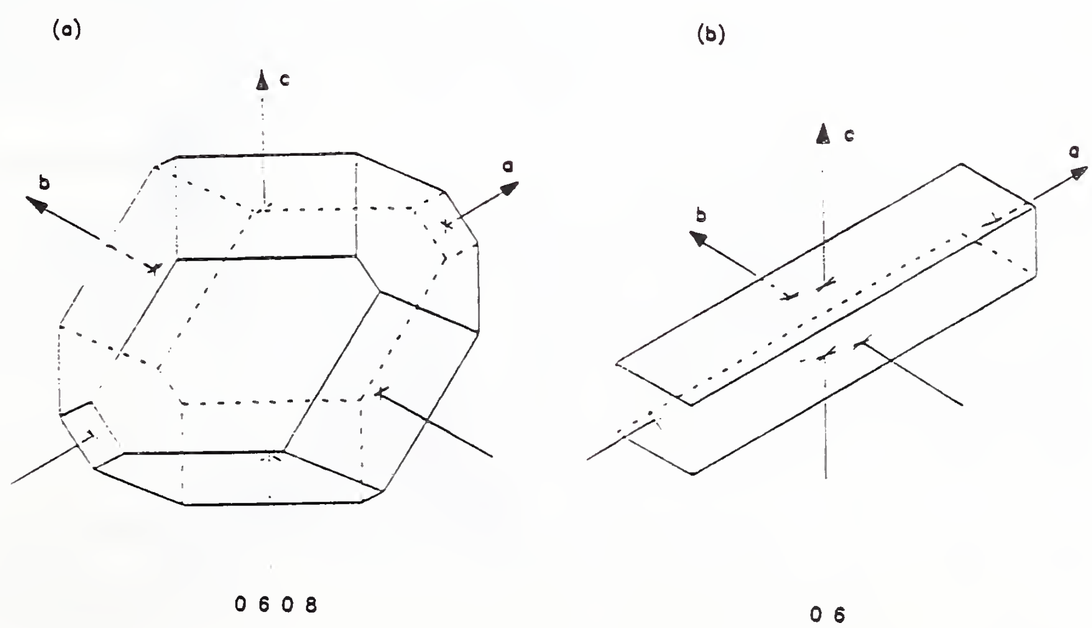


Fig. 3. The Wigner-Seitz polyhedron around the Cu(1) atom in the high T_C $YBa_2Cu_3O_7$, calculated using (a) metallic and (b) ionic radii.

Fig. 4.

Comparison of the Mössbauer spectra from two samples of $RBa_2(Cu_{0.97}Fe_{0.03})_3O_{7-x}$ for two different rare earths (R=Pr and R=Er) with x≈1. The outer two lines observed for R=Pr (but not for R=Er) are due to the presence of a magnetic hyperfine field and arise from iron atoms substituted on Cu plane sites. These results are consistent with the view that a superconductor is not formed for R=Pr because the Cu atoms on the planes in this system form an antiferromagnetic structure.

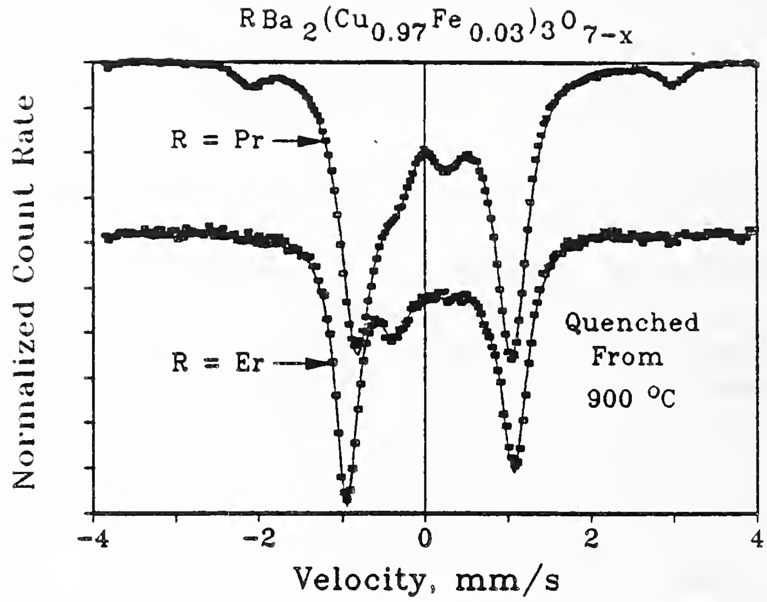
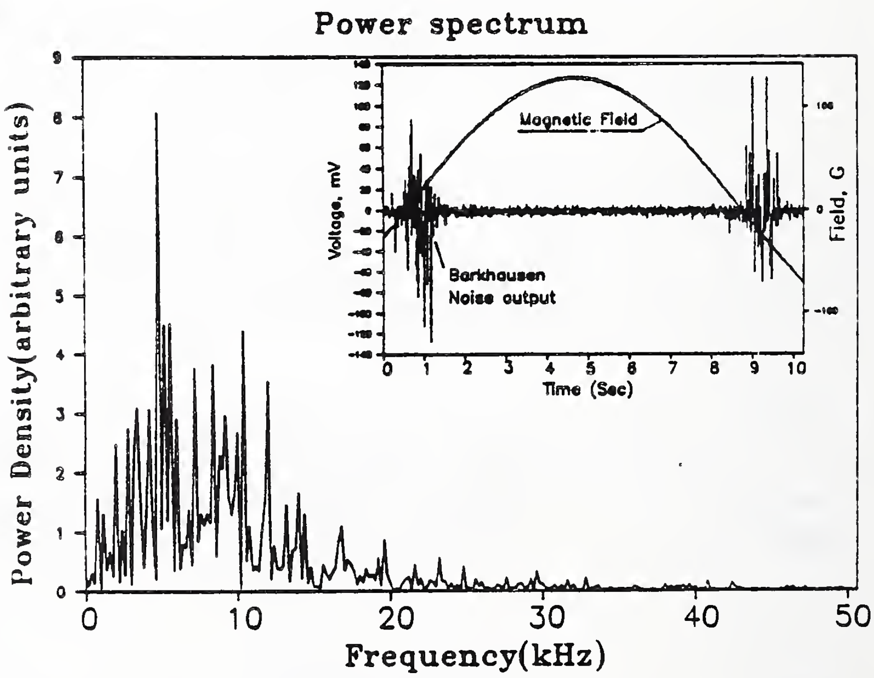


Fig. 5.

Field Emission Scanning Electron Microscope (FESEM) high magnification picture (250,000X) of the surface of a $Ag+Fe_3O_4$ nanocomposite containing ~30 wt.% Ag showing the nanometer-sized regions of silver (light) and iron oxide (dark) in the thin-film deposit. For this high magnification picture no special specimen preparation was required.

Fig. 6.

Power spectrum of Barkhausen Noise from 20mg sample of Fe foil measured at room temperature. The power density spectrum was obtained from the digitized noise (as shown in the inset) by taking the autocorrelation of the fast fourier transform.



HIGH TEMPERATURE MATERIALS CHEMISTRY John W. Hastie

The research program in high temperature materials chemistry emphasizes the thermodynamic, chemical-kinetic, interfacial microstructure, and molecular-level behavior of inorganic materials in high temperature process and service environments. Specific current objectives are (1) to support the U.S. Steel Industry through development of a thermodynamic database and solution model for prediction of slag, refractory, and inorganic steel inclusion thermochemistry; (2) to develop and apply (eg., to refractory ceramics and composites) a new molecular-specific methodology for obtaining thermal and chemical stability data at ultra-high temperatures (2000 - 5000°C) for the design of hypersonic transport vehicles and various defense applications; recent surveys by NMAB and Industry have indicated a critical need for thermochemical and other materials property data at these temperatures; (3) to develop process models and basic data for the preparation of superconducting films using laser or other vapor deposition techniques; (4) to enhance the utility of superalloy and carbon/carbon composites in high temperature oxidation environments through mechanistic studies of oxidation; (5) to obtain phase equilibria, kinetic, and mechanistic data for the development of stored chemical energy propulsion systems; and (6) to provide technical support to the NIST-ACerS Ceramic Phase Diagram Data Program through critical evaluation and modeling of phase diagrams and the development of computer graphics for computer storage and manipulation of phase diagrams.

FY 88 Significant Accomplishments

- A thermodynamic model and accompanying database have been developed for the prediction of detailed composition and phase properties of multicomponent-multiphase oxide systems. The model has been shown to be applicable to typical iron-making slags and refractories used in steel processing. The model database currently contains over 125 phase components and has been successful in predicting high temperature thermodynamic activities and phase compositions in mixtures containing up to eight component oxides. Several industries are beginning to substitute this model for the overly simplistic chemical models currently used in transport-type processing computer codes.

- The laser-induced vaporization mass spectrometric technique (developed in-house) has been applied to vapor plumes produced from superconducting $YBa_2Cu_3O_x$ targets. Time resolved mass and optical emission spectra were obtained for many neutral and ionic species in the laser generated plume, including bimetallic species. Work is underway to correlate the plume species with the composition and superconducting characteristics of thin films prepared by laser ablation of superconducting targets. Ultrafast signal averaging (100 MHz) and optical multichannel spectroscopic capabilities have been added to the laser-induced vaporization facility. These new techniques permit both mass and optical spectrometric techniques to be

applied to the study of non-equilibrium processes occurring on the laser-impact time scale.

- A new mass spectrometric and complementary thermochemical investigation of the ternary Nb-Al-O system has been initiated as part of a cooperative DARPA program with CEBELCOR (M. Pourbaix, Belgium) and the University of Florida to develop oxidation models of advanced intermetallics. Activity data on Nb-Al intermetallics and their oxidation products are being obtained under this study. Also a preliminary THERMOCALC model of the Nb-Al-O system has been developed to guide the experimental work.

- Mass spectrometric investigations of the reactions of high energy oxidants (F_2 and ClO_3F) with Li-Mg-Al alloys, aluminum, and alumina have been undertaken in support of the development of new stored chemical energy propulsion devices by U.S. Industry and DOD. Evidence for kinetic inhibition of the reactions by formation of oxide or fluoride protective layers has been found. These effects will have to be taken into account in development models of successful devices.

- A PC-based computer program for the retrieval and manipulation of phase diagrams has been developed and released for beta-testing by industrial and other agency sponsors of the joint NIST-ACerS Phase Diagrams for Ceramists Data Program. The graphics database and the program to access it are the result of a long standing collaborative program between the High Temperature Materials Chemistry Group and the American Ceramic Society.

<u>Steel Slag - Refractory Thermochemistry</u>
J. W. Hastie, E. R. Plante, D. W. Bonnell, W. S. Horton[1]

[1]Guest Scientist

Modeling and complementary experimental work has continued on the development of a generic predictive model of iron and steel-making slag and refractory thermochemistry. The computer model is already in use by several steel and other industrial laboratories and consortia. In addition to its present utility for existing steel-making technology, the model is designed to be applicable to new direct reduction steel making technologies being planned or under development.

Recent measurements have been made to determine the alkali oxide activity in a blast furnace slag sample supplied by Inland Steel Co. These data were obtained using the Knudsen effusion modulated-beam mass spectrometric system. In addition, as a check on the blast furnace slag measurements, baseline experiments were conducted using reference basic (high CaO) glasses with known amounts of alkali. The data are the first measurements on basic slags and will be used to verify and extend the model calculations. The results included the K(g), Na(g), and O_2(g) partial pressures as a function of temperature and condensed phase composition.

The computer model is quite general and has had excellent success in modeling a variety of highly complex slags. In application to basic steel-making slags, we have encountered discrepancies in experimental versus model results, which are due to uncertainties in the available thermochemical data. In particular, available Gibbs energies of formation data for the calcium aluminosilicate liquids in the database (i.e. $CaAl_2Si_2O_8(l)$ {anorthite}, $CaAl_2Si_4O_{12}$ {calcium leucite}, and $Ca_2Al_2SiO_7(l)$ {gehelenite}) are currently being refined. In addition, although thermodynamic data for mixed alkali silicates {$KNaSiO_3(l)$ and $KNaSi_2O_5(l)$} are unavailable, mixed alkalis are known to be significant vapor species above mixed alkali systems which implies the existence of a configurational stability (predominately entropic at high temperature) for mixed alkalis. Trial species functions have been introduced into the database and are being tested.

With the expansion of the database to over 150 components covering 18 elements, it has become necessary to develop methods for handling the database via computer-aided data preparation tools, and permit broader ranges of temperatures. This has resulted in a new development effort which includes modifying the equilibrium code SOLGASMIX to permit automated database use and refitting the entire current database to expand the range of validity of the thermodynamic functions.

Thermodynamic and Kinetic Stability of Refractory Materials at Ultra-High Temperatures

D. W. Bonnell, P. K. Schenck, J. W. Hastie

The coupling of laser heating with mass spectrometric analysis has the potential for providing quantitative thermochemical and other data for refractory materials at temperature and pressure extremes heretofore inaccessible to standard techniques. In addition, degradation of materials by high powered lasers is important in, for example, the design of laser fusion processes, laser welding, laser processing of ceramics (most recently, superconducting and diamond films), laser etching of semiconductor components, laser annealing of surface alloys, and in the durability of refractories in defense and space applications.

We have shown that a Nd/YAG laser system focused to power densities in the region of 10^8 W/cm^2, is a convenient energy source for producing controlled vapor plumes with generally negligible post-vaporization perturbation of the neutral species identities and concentrations. The NIST-LIVMS technique utilizes time-resolved mass analysis to provide time-of-flight species specific information on temperature, ionic and neutral precursors, and time history of the laser heating process. Figure 1 shows the time-of-flight signals for some of the carbon species emitted from a graphite target. These multimode signals exhibit both the complexity of the time-of-flight signals and the need for ultrafast data acquisition and signal averaging. To meet this need a 100 MHz signal averager has been added to the LIVMS. Earlier studies of the BN and graphite systems have been extended to include carbide targets. SiC targets were vaporized and studied by the LIVMS technique.

Evidence for polymeric $(SiC)_2$ evolution was found in addition to the expected decomposition vaporization.

Mechanistic Determination of Superconducting Thin Film Deposition
P. K. Schenck, D. W. Bonnell, J. W. Hastie

A joint project with the Magnetic Materials group was initiated this year to apply the LIVMS technique to high temperature superconducting targets. Thin films deposited by laser vaporization from bulk high temperature superconducting material have shown evidence of high temperature superconductivity. Time resolved mass spectra were obtained for many neutral and ionic species in the laser generated plume from $YBa_2Cu_3O_x$ targets, including bimetallic species CuBa and YCu. Observation of molecular O_2 in the plume is consistent with the oxygen deficiency of laser deposited films reported by other researchers. This deficiency requires that the films be annealed at high temperature in O_2 to restore the proper stoichiometry for high temperature superconductivity. A high speed optical multichannel analyzer was added to the LIVMS facility to allow optical spectroscopic techniques to be applied to the study of the laser generated plumes. Figure 2 shows the optical emission spectra from the $YBa_2Cu_3O_x$ plume during the initial vaporization and after 5000 laser shots. The spectra indicate that the relative amounts of Y and Ba in the plume are lower after several thousand laser shots. This result suggests that the ablation surface must be kept fresh to maintain the superconductor Y-Ba-Cu stoichiometry in the plume. Work is underway to further correlate plume species with the composition and characteristics of thin films of ceramic superconductors.

Processing and Protection of High Temperature Structural Materials
J. W. Hastie, D. W. Bonnell, E. R. Plante, U. Kattner[1]

[1]Guest Scientist, Metallurgical Processing Group

This activity represents a new group thrust initiated late in FY 88 under a DARPA sponsored program joint with the University of Florida. An industrial panel advises the program.

The purpose of this effort is to combine existing thermochemical data with new experimental determinations and modeling formalisms to develop data-banked information and models which provide for improved design and evaluation of refractory superalloy and composite systems. Niobium alloys and particularly the Nb-Al-O system are being investigated initially. Data are to be obtained on the mechanisms of oxidation, species transport rates, thermochemical stabilities, activities, vapor phase interactions, solubilities, and diffusivities for the development of reliable models of coating/substrate/gas interactions. In the beginning stages, most of the work has concentrated on the review of sparsely available literature data and on the development of a heuristic ternary phase diagram to use as an experimental guide both for NIST and for the University of Florida groups.

In the Nb-Al-O system intermediate condensed-phase compounds exist: the binary compounds Nb_3Al, Nb_2Al, $NbAl_3$, NbO, NbO_2 and Nb_2O_5 and, along the

quasibinary Al_2O_3-Nb_2O_5 section, four ternary compounds $49Nb_2O_5 \bullet Al_2O_3$, $25Nb_2O_5 \bullet Al_2O_3$, $9Nb_2O_5 \bullet Al_2O_3$ and $Nb_2O_5 \bullet Al_2O_3$, with the latter two having significant ranges of homogeneity. For the phase diagram calculation of this system, the thermodynamic quantities for some phases are unknown or not enough data are available to determine them, simplifications and assumptions have been made in the current calculation.

<u>Stored Chemical Energy Systems</u>

E. R. Plante, D. W. Bonnell, L. P. Cook[1]

[1]Ceramics Division

Mass spectrometric determinations of the reaction products of potential oxidants [$ClO_3F(g)$ or $F_2(g)$] with reactive metals or alloys (Li, Mg, Al) have been made as part of an ongoing propulsion program sponsored by ONR. The reaction of ClO_3F with Li-Al or Li-Al-Mg alloys was observed to form gaseous Li and Al mixed halides, which need to be included in thermochemical models of the combustion process. Under laboratory steady state conditions, the Cl_2 and O_2 gas pressures, from ClO_3F decomposition, were found to be much greater than for F_2 indicating preferential fluorination of the alloys. However, there is no evidence for reaction of oxygen with the alloy under low pressure (10^{-5} atm) laboratory experimental conditions. Under higher pressure conditions (0.3 atm) reaction with oxygen takes place erratically as evidenced by large fluctuations in the oxygen pressure. Aluminum oxide reacts with $F_2(g)$ to form $AlF_3(g)$ and $O_2(g)$, while the addition of Al metal produces mainly $AlF(g)$. Some evidence of kinetic inhibition of the reactions by formation of oxide or fluoride protective layers was observed. Future work is being directed toward studying the reactions of liquid organic perfluoro compounds such as perfluoro tetrahydrofuran which has been shown to react with Al to form $AlF(g)$ and $CO(g)$. The use of organic fluorides extends the advantages of safety and high density to the propulsion system.

<u>Phase Diagram Graphics, Evaluation, and Modeling</u>

P. K. Schenck, J. W. Hastie, J. R. Dennis[1]

[1]ACerS Research Associate

New computer software has been developed to handle complex binary and ternary diagrams using stand alone desk top computers in support of the Phase Diagrams for Ceramists Data Program. The publication-ready diagrams for the recently completed Vol. 6 of "Phase Diagrams for Ceramists" were generated with this system. Software was also developed to provide PC-access to the ceramics phase diagram graphics database. PC-users will have the ability to retrieve data from the screen display in a choice of user units (e.g., °C, °F, or K). Mixture compositions in binary phase fields can be determined interactively by use of the lever rule and the results displayed on the PC-monitor screen. The PC-based program and the graphics database for Vol. 6 have been distributed for beta testing to the technical representatives of the more than twenty industrial sponsors of the joint NIST/ACerS phase equilibria program. Figure 4 shows the title screen from the distributed PC software and a typical phase diagram as displayed on the PC monitor screen.

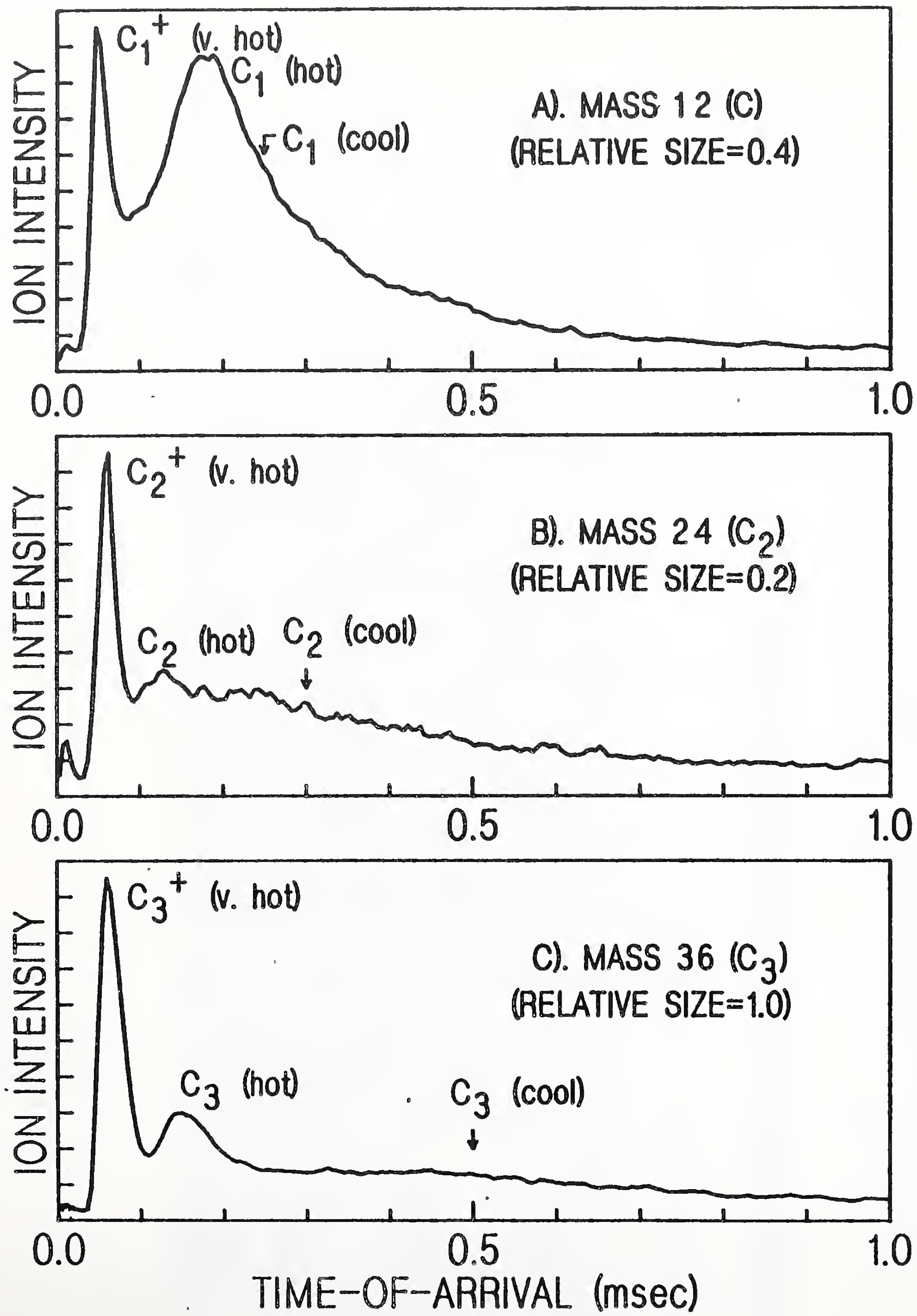


Figure 1. Mass and time resolved abundance profiles for neutral and charged species, sampled from a laser produced graphite plume

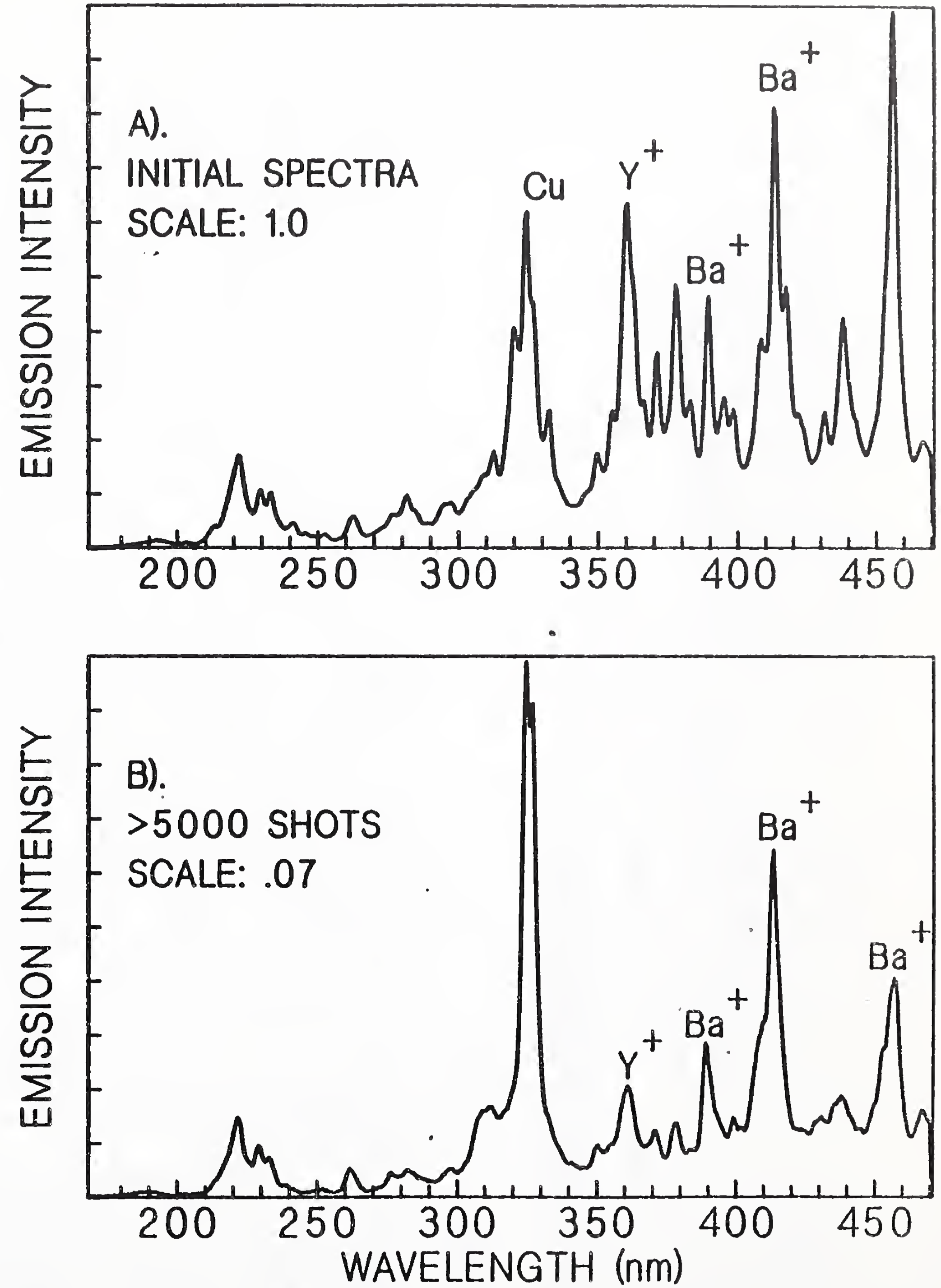


Figure 2. Wavelength and time resolved optical emission spectra for a laser plume produced from a superconducting composition of $YBa_2Cu_3O_x$

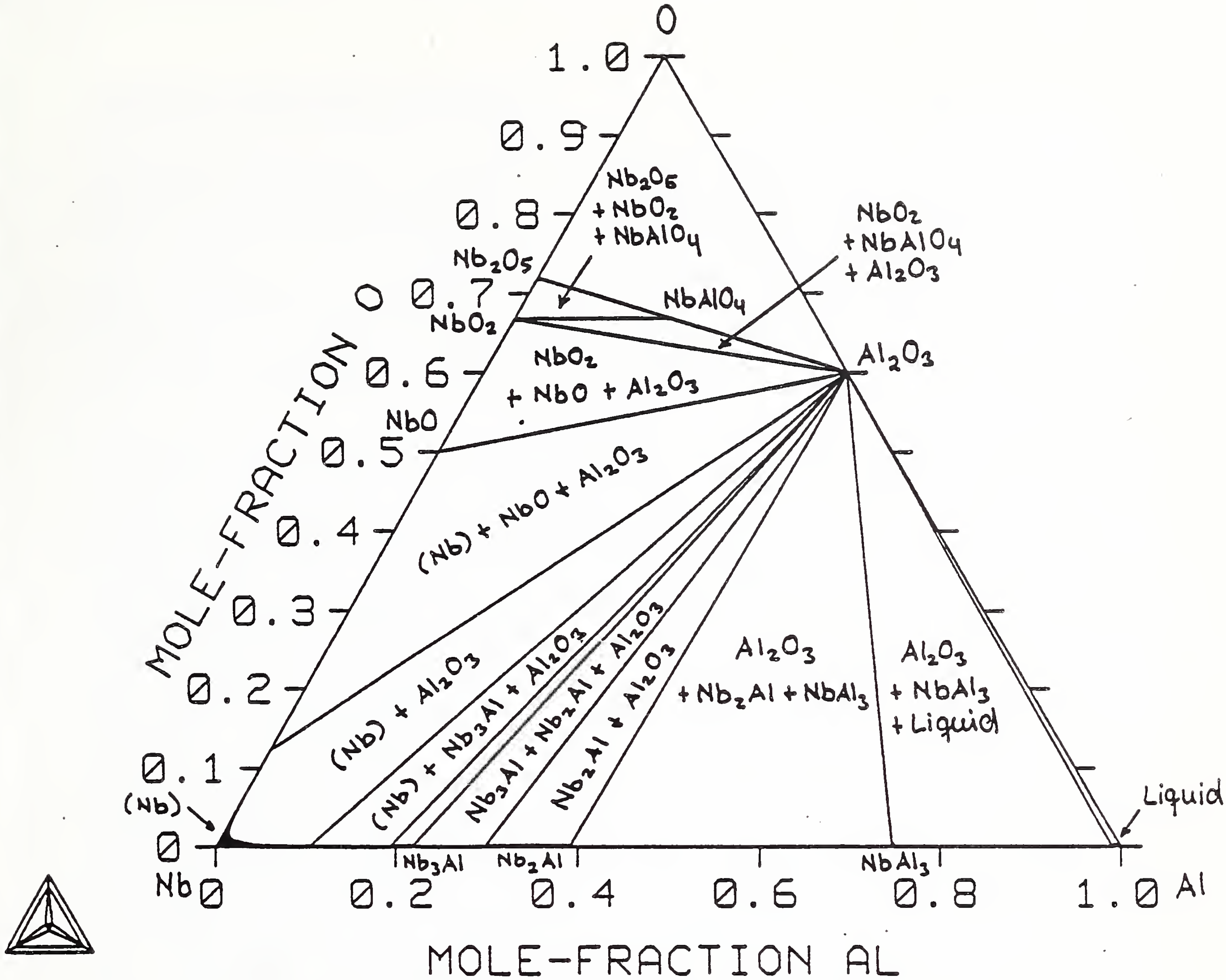


Figure 3. ThermoCalc-based preliminary ternary phase diagram of the Nb-Al-O system; isothermal section at 1400°C

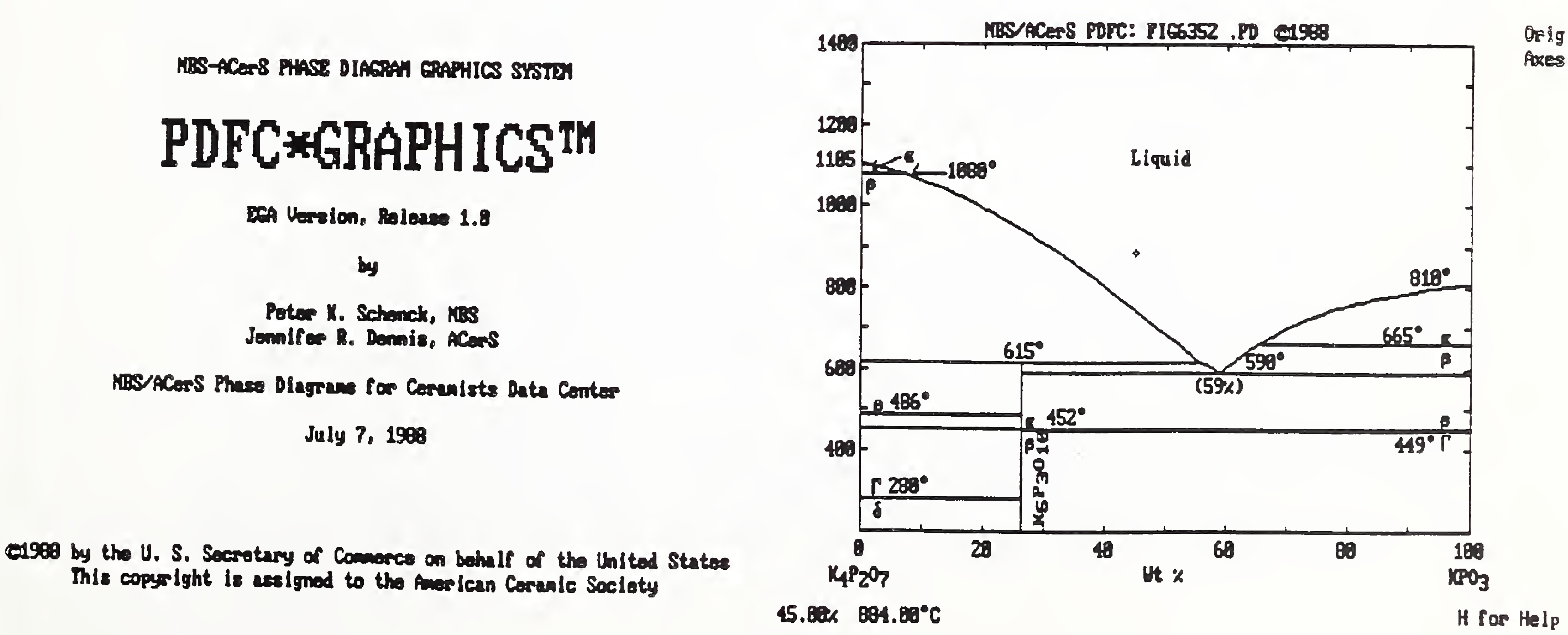


Figure 4. Typical screen display of a PC-accessible phase diagram

MECHANICAL PROPERTIES OF METALS John H. Smith

The Mechanical Properties of Metals Group was formed during FY 88 by combining personnel transferred into the Metallurgy Division from the Fracture and Deformation Division with personnel in the Metallurgy Division responsible for the microscopic characterization of metals. The mission of the group is to: (1) characterize the mechanical and physical properties of metals and metal based materials (2) develop improved test methods for characterizing the mechanical properties of metals (3) evaluate the structural integrity and durability of metallic structures and components and (4) characterize the microstructure and composition of metals and alloys.

The activities of this group are consist of (1) research on the mechanical properties of metals (2) research on test and calibration methods (3) structural characterization of materials (4) investigative metallurgy (failure analysis) and (5) technology transfer. The research on the mechanical properties of metals consists primarily of special tests to evaluate the properties of newly developed materials and on tests to evaluate the structural integrity of metallic components and structures. Most of this work is supported by other government agencies and involves close collaboration with industry associations such as the American Association for Railroads and the Compressed Gas Association and with several individual companies. The research on test methods and calibration methods is largely supported by NIST funds and is directed at improved methods for characterizing the properties of both conventional materials and of new advanced materials. The structural characterization of materials involves the development of new techniques and the use of optical and electron microscopy, image analysis and X-ray analysis to evaluate the microstructure and composition of metal and alloys. This activity is carried out in collaboration with and in support of the most of the research being carried out in metals processing and advanced material in the Division and in IMSE. The investigative metallurgy (failure analysis) projects are all carried out at the request of and with funding from other agencies and outside organizations. These activities lead to a better understanding of how our metallic structures and components perform in service and frequently lead to improvement in design codes and materials standards. This group's most significant activity in technology transfer has been providing the direction and operation of the Mechanical Failure Prevention Group (MFPG). For over 25 years, the MFPG has very successfully organized an annual or semiannual conference and published a proceeding on the analysis, detection, and prevention of failures in a wide variety of mechanical components.

FY 88 Significant Accomplishments

- An extensive investigation was completed to determine the cause of the structural collapse of a large oil tank owned by the Ashland Oil Company. Actions were identified to reduce the probability of similar occurrences in other large, above ground tanks.

- A major investigation was completed for the Federal Railway Administration to determine the mechanical properties and fracture properties of steels used in railroad tank cars. The results of this investigation will permit accurate determination of critical flaw sizes in tank cars to be determined.

- An investigation of the effects of heat treatment on the mechanical properties and fracture toughness of HSLA steels or use in naval vessels was completed.

- A series of investigations to determine the causes of failure of components in electric power generation was completed.

- The existence of a new form of sustained load cracking in aluminum pressure vessels was confirmed and procedures were developed to inspect these vessels.

- The 42nd meeting of the Mechanical Failures Prevention Group (MFPG) entitled "Technology Innovation -Key to International Competitiveness" was organized and held. The proceedings of the 41st meeting of the MFPG entitled "Detection, Diagnosis and Prognosis of Rotating Machinery to Improve Reliability, Maintainability, and Readiness through Application of New and Innovative Techniques" was published. The 43d meeting of the MFPG entitled "Advanced Technology in Failure Prevention" was held in San Diego Cal.

- A new procedure was developed to use infrared thermography to study crack initiation and propagation during tensile and fatigue loading in aluminum and steel.

- A new method for using the electron microprobe for the quantitative analysis for determining sodium in zinc was developed.

- A study of the formation of high temperature thin film superconductors on silicon wafers was completed.

- An electron microscope study of the properties of thin film silver-iron oxide granular metals was completed.

Mechanical Properties Research
G. E. Hicho, J. H. Smith, and T. R. Shives

The mechanical properties of metal research emphasizes the evaluation of newly developed materials and the evaluation of structural integrity of metallic components and structures. Increasingly the emphasis is on the evaluation of advanced metallic materials. Nearly all of this research is funded by other government agencies and generally involves close collaboration with the producers and or users of the materials.

Characterization of Railroad Tank Cars Steels - Extensive research has been conducted to evaluate the mechanical properties, fracture resistance and crack arrest properties of presently used and new steels for the construction of railroad tank cars used to transport hazardous materials. Presently used, as-rolled steels are subject to fracture under some operating conditions when the tank cars are involved in accidents. However, newly developed microalloyed steels and new heat treatments of present steels, such as

normalizing and stress relieving reduce the possibility of fracture. The effect of composition, temperature, and strain rate on the mechanical properties and fracture resistance of the steels have been determined. The dynamic mechanical properties of AAR M128 and ASTM A212B steels were determined using Charpy impact tests and dynamic tear tests. The transition temperature, crack initiation energy, crack propagation energy, and total fracture energy were determined. For these steels, which are used in currently existing tank cars, the ductile to brittle transition temperature was found to be in the range of temperature at which the tank cars are often used. A review and evaluation was made of new steels for tank cars and of the feasibility for tank car applications of new microalloyed and controlled steels based on a comparison of the fracture properties of the new steels with presently used steels. The fracture toughness of ASTM A-212 and AAR TC-128B in the normalized condition was evaluated as function of temperature to permit a determination of the critical crack size in railroad tank cars to be estimated. Additional research was initiated to determine the crack arrest properties of normalized TC-128B, controlled rolled A808 steel, and microallyed A710 steel.

Evaluation of HSLA-80 Steels - The effect of heat treatment on the mechanical properties of newly developed HSLA-80 steel was determined under a program sponsored by the Navy to determine the feasibility of using these steels for ship construction. HSLA-80 is a precipitation hardening ferritic steel containing about 1.2 percent copper for strengthening and niobium for grain size control.

Safety Design Criteria for High Strength Steel Cylinders - Research is carried out under sponsorship of the Department of Transportation Office of Hazardous Materials to establish a sound, technical basis for safety standards governing the safe design, manufacture, and use of seamless pressure vessels (cylinders) for the storage and transportation of compressed gases. Present research is aimed at developing a design criteria for the construction of high strength steel cylinders to prevent failure by fracture or by stress corrosion. Collaborative test programs with Taylor Wharton Inc., Norris Cylinder Inc., and T.I. Chesterfield Ltd. are being conducted to establish a fracture criteria that accurately predicts the fracture behavior of thin walled, ductile steel cylinders. Results of this investigation have led to a preliminary design specification for higher strength steel cylinders.

Sustained Load Cracking in Aluminum Cylinders - A unique form of sustained load, intergranular has been found in the neck and thread area of type 3AL aluminum cylinders made from type 6351 alloy. Collaborative investigations are being carried out with Luxfer USA, Alcan Ltd., and Walter Kidde Inc. to determine the extent of this form of cracking and to determine which cylinder designs are subject to fracture and which are subject only to leaking without rupture. Results of this investigation have resulted in changing from type 6351 alloy to 6061 alloy for the majority of the of new cylinders produced in the United States.

Evaluation of Aramid Fiber Rope - Under sponsorship of the U.S. Coast Guard, the effect of service life on the mechanical properties or aramid fiber rope

used to support large radio transmitting towers was evaluated. The tensile strength, fatigue strength, creep, and stress relaxation of ropes were evaluated before and after exposure to ocean environments. No apparent degradation due to the environment was found.

Research on Test and Calibration Methods
T. R. Shives, G. E. Hicho, J. H. Smith, and Y. Huang*
* Guest Scientist, Institute of Metal Research, Academia Sinica, China

Macrohardness Test Block Standards - Under partial support from the NIST Office of Standard Reference Materials and in collaboration with the ASTM Committee E-28 on Mechanical Testing to conduct an intercomparison of Rockwell hardness all test blocks and indentors that are marketed in the United States. The indentors have been characterized by interferometry and with an optical comparator. Preliminary results of the Rockwell Brale indentors evaluation have shown substantial variation from the indentor geometry required by the ASTM specification. This is believed to be the cause of much of the variability shown in the hardness test results as shown in Figure 1.

Infrared Studies of Crack Growth - A new thermographic method based on the measurement of infrared (IR) emission from the surface of a loaded body was developed to study the cooling and heating in aluminum and steel specimens during tensile and cyclic loading. A typical thermogram is shown in Figure 2. This thermogram was used to measure the temperature at any point on the body relative to the reference temperature T. Figure 2 shows the dynamic process of the infrared emission for a carbon steel specimen, with a center-cut hole, subjected to tensile loading. Prior to yielding, the IR emission shows that cooling was concentrated in the area of the crack tip. After yielding, the IR emisson shows that heating the deformed specimen occurs at the crack tip. The characteristic "butterfly" shape of the plastic zone was observed to extend immediately to the boundary of the specimen (see image F in Fig. 2). In Figure 2, the point P_T corresponds to the inflection point marking the boundary between the IR cooling and heating emission regions. This value is identified as the thermoelastic limit load and is lower than the 0.2% offset yield strength. Similar tests conducted on fatigue specimens showed comparable results. In the deformation of metals, the measurement of this IR cooling emission technique effect could be used to detect the stress concentration area.

Fracture Toughness of Aluminum using IR and DC Potential Drop Techniques - A new measurement technique was developed to measure crack iniation and extension during fracture testing. J-integral fracture toughness tests were conducted at room temperature on aluminum three-point bend and compact tension specimens. The ductile-crack-resistance curves were measured using the multiple specimen method. Crack growth was automatically recorded using both the potential drop and infrared (IR) vibro-thermographic techniques. The IR vibro-thermographic technique made it possible to obtain real-time measurements of both crack initiation and extension during the entire fracture toughness test.

Certification of Ferrite in Weld Standard - A ferrite in weld Standard Reference Materials (SRM) has been prepared and is ready for certification. This material is composed of 10% 430 stainless steel (ferritic) blended with 90% 310 stainless steel (austenitic). Powder metallurgical techniques are used to prepare these materials. The materiasl are certified by determining the percent ferrite in each specimen image and x-ray fluorescence analyses. The certified standard reference materials will be available calibrate x-ray diffraction equipment that is used in the determination of ferrite in welds.

Structural Characterization
A. J. Shapiro and L.C. Smith

The structural characterization activity provides the management of the optical microscopy, electron microscopy, and X-ray facilities for the Metallurgy Division. Most of the activity is conducted in close collaboration with the other research projects in the Division such as metals processing, magnetic materials, metal matrix composites, and other advanced materials. The emphasis has increasingly shifted to the microstructural characterization of advanced materials and to developing improved procedures and techniques to characterize the structure of these materials. Examples of this work are the investigation of grain boundary migration in MgO-CaO-ZrO, the evaluation of the structure of thin film Ag-Fe oxide granular metals, and the structural characterization of thin film high temperature superconductors. A new method of quantitative X-ray analysis has been developed to determine the amount of sodium present in alloys of zinc.

High temperature thin film superconductors of YBa_2Cu_3On were fabricated by sputter deposition. The effect of process variables such as substrate temperature, target to substrate angle and the partial pressure of oxygen in the sputtering atmosphere on the final composition of the thin film superconductor were determined by X-ray microanalysis.

Composite materials made from nanometer sized particles of metals (e.g. Ag) and nonmetals (e.g. FeO) are referred to as granular metals. When the metallic consistutent has a strong magnetic moment, the granular metal composite is found to have significantly improved magnetic properties compared with the homogeneous form of the same metals. An extensive electron microscope study was undertaken to evaluate the microstructure of these granular metals and establish relationships between the microstructure and the unique magnetic properties of these materials.

Investigative Metallurgy
J. H. Smith, G. E. Hicho and T. R. Shives

These investigations are done at the request of and funded by other government agencies or Congressional Committees. These investigations apply our expertise in the characterization of metals to determine the cause of failure and to evaluate the structural integrity of metallic components and structures. The results of these investigations lead to improvement in the selection and use of materials and to improvement in design and safety codes and standards.

Ashland Oil Tank Failure - A significant part of the Group's effort for this year was devoted to a major investigation jointly conducted with the NIST Center for Building Technology to determine the cause of the catastrophic collapse of a large (4 million gallon) oil storage tank. The collapse of the tank was caused by brittle fracture originating from a preexisting defect (Fig. 3a) in the wall of the tank. The material around the defect was embrittled by heating during the dismantling and reconstruction of the tank. This resulted in reduction in the fracture toughness of the metal surrounding the defect and the combination of low temperature at the time the tank was first filled and the stress created by the oil in the tank was sufficient to initiate the fracture. As shown in Figure 3b, the tank was being operated at a temperature substantially below the nil-ductility-temperature (NDT) and consequently the fracture did not arrest and total collapse of the tank occurred. The results of this investigation have led to continuing collaboration with the Environmental Protection Agency and with the American Petroleum Institute to develop improved design codes, construction standards, and inspection standards for above ground storage tanks.

Failure Examinations for the Potomac Electric Power Company

During the past year we have presented the Potomac Electric Power Company (PEPCO) 18 reports on failures that occurred in their power generating equipment. Our failure analyses consisted of a variety of examinations that included weld, waterwall, and bolt failures. The results of these investigations are used by PEPCO to correct operating conditions and prevent future failures of the same type which reduces down time for the power generating equipment during peak power demands.

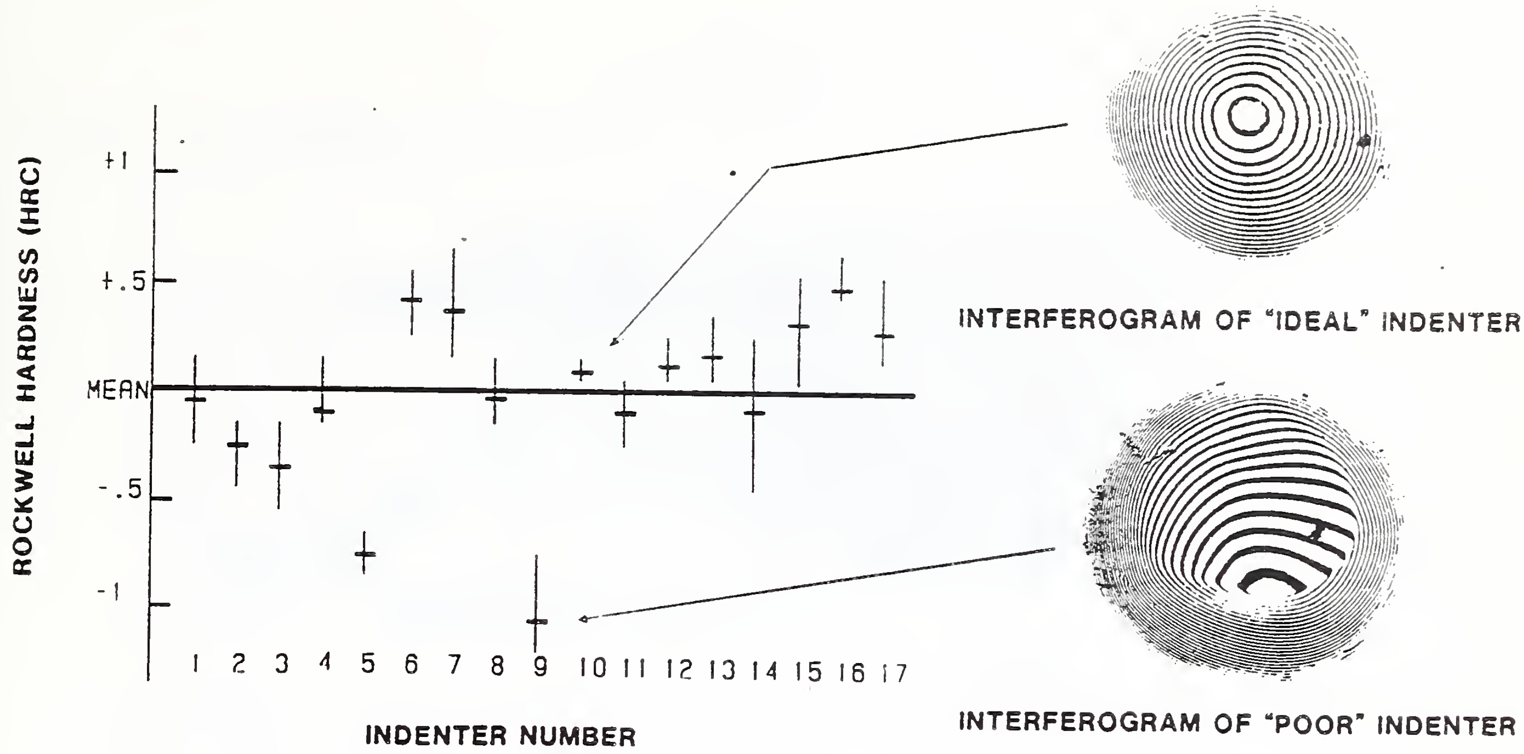


Figure 1. Effect of Rockwell C (Brale) Indentor Geometry on Variability of Hardness Measurement.

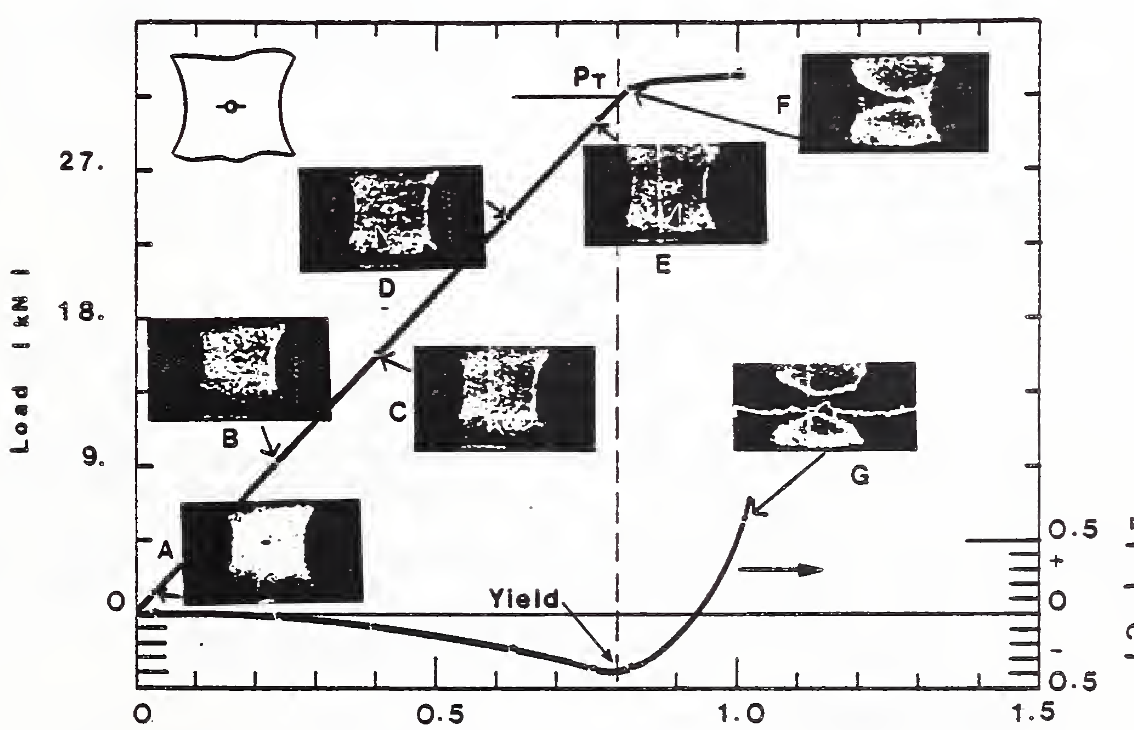


Figure 2. Temperature Distribution at the Tip of a Crack During Fracture of a Center Cracked Wide Plate.

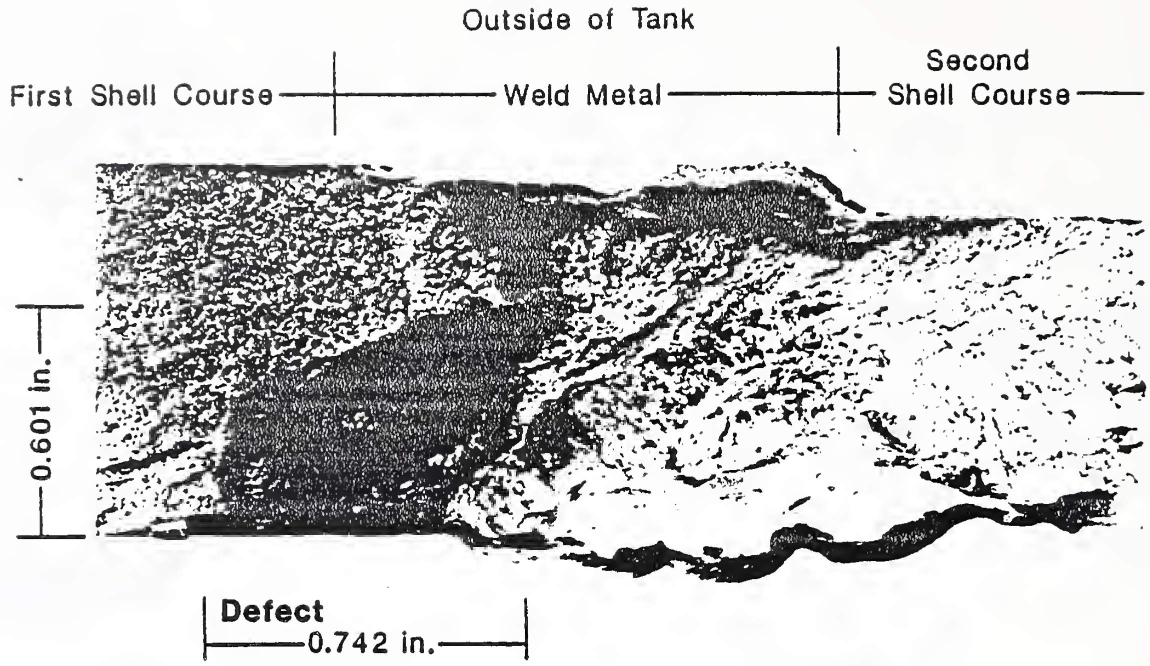


Figure 3a. Defect on Fracture Surface from which Tank Failure Originated.

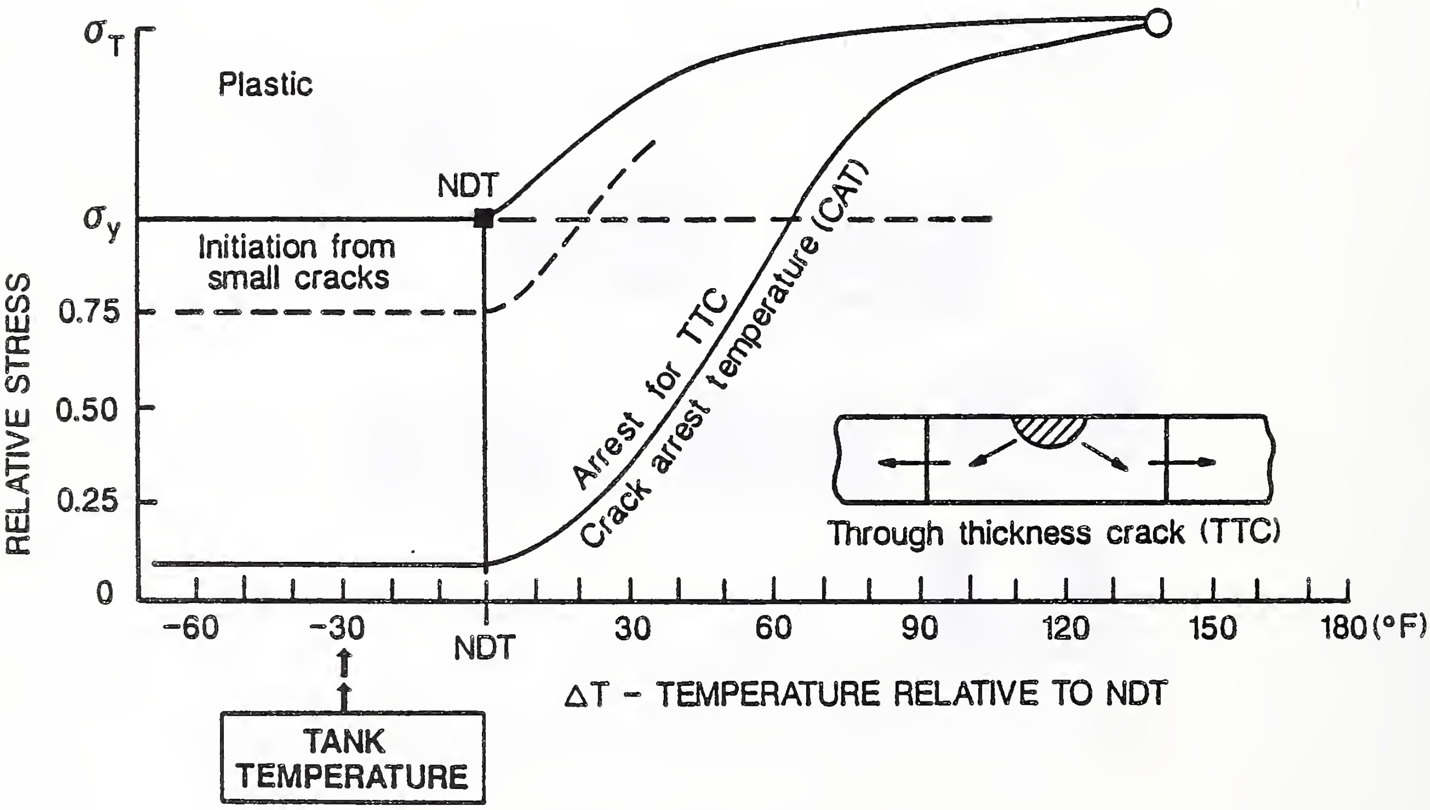


Figure 3b. Fracture Analyses Diagram (FAD) showing that Tank was Operating well below NDT Temperature.

PUBLICATIONS

Metallurgical Processing Group

Bendersky, L. A.; "Al-Mn μ Phase and Its Relation to Quasicrystals." J. of Microscopy, 146, 303 (1987).

Bendersky, L. A.; "Quasicrystals and Quasicrystal-Related Phases in the Al-Mn System." J. of Mat. Sci. and Eng., 99, 331 (1988).

Bendersky, L. A.; "Structural Relationship Between Crystalline and Quasicrystalline Phases in Al-Mn System." Proceedings of International Workshop on Quasicrystals; Mater. Sci. Forum, Vols. 22-24, 151 (1987).

Bendersky, L. A.; Biancaniello, F. S.; Schaefer, R. J.; "Amorphous Phase Formation in $Al_{70}Si_{17}Fe_{13}$ Alloy." J. Mat. Res., 2 (4), 427 (1987).

Bendersky, L. A.; Graves, J. A.; Biancaniello, F. S.; Perepezko, J. H.; Boettinger, W. J.; "Pathways for Microstructural Development in TiAl." Mat. Sci. and Eng., 98, 265 (1988).

Bendersky, L. A.; Kaufman, M. J.; Boettinger, W. J.; Biancaniello, F. S.; "Solidification of an 'Amorphous' Phase in Rapidly Solidified Al-Fe-Si Alloys." J. of Mat. Sci. and Eng., 98, 213 (1988).

Bendersky, L. A.; McAlister, A. J.; Biancaniello, F. S.; "Phase Transformation During Annealing of Rapidly Solidified Al-based Al-Fe-Si Alloys." Met. Trans. A; in press (1988).

Bendersky, L. A.; Voorhees, P. W.; Boettinger, W. J.; Johnson, W. C.; "The Role of Elastic Energy in the Morphological Development of a Ni-Ti-Al Alloy." Scripta Met., 22, 1029 (1988).

Bennett, L. H.; Cahn, J. W.; Schaefer, R. J.; Rubinstein, M.; Stauss, G. H.; "Icosahedral Symmetry vs. Local Icosahedral Environment in Al-Mn Alloys from NMR." Nature, 326, 372 (1987).

Blendell, J. E.; Handwerker, C. A.; Vaudin, M. D.; Fuller, E. R., Jr.; "Composition Control of the Microstructure of $Ba_2YCu_3O_{6+x}$." J. of Cryst. Growth, 89, 93 (1988).

Boettinger, W. J.; "Microstructural Variations in Rapidly Solidified Alloys." Mat. Sci. and Eng., 98, 123 (1988).

Boettinger, W. J.; Bendersky, L. A.; Biancaniello, F. S.; Cahn, J. W.; "Rapid Solidification and Ordering of B2 and L21 Phases in the NiAl-NiTi System." J. of Mat. Sci. and Eng., 98, 273 (1988).

Boettinger, W. J.; Bendersky, L. A.; Schaefer, R. J.; Biancaniello, F. S.; "On the Formation of Dispersoids during Rapid Solidification of an Al-Fe-Ni Alloy." Met. Trans. A, 19A, 1101 (1988).

Boettinger, W. J.; Coriell, S. R.; Trivedi, R. K.; "Application of Dendritic Growth Theory to the Interpretation of Rapid Solidification Microstructures." Rapid Solidification Processing: Principles and Technologies IV; R. Mehrabian, P. A. Parrish, eds.; Claitor's Publishing Division; Baton Rouge, LA; 13 (1988).

Burton, B. P.; "Application of the ϵ-G Approach to Binary Rhombohedral Carbonate Systems." Proceedings of CALPHAD-XVII; in press (1988).

Burton, B. P.; Davidson, P. M.; "Multicritical Phase Relations in Minerals." Advances in Physical Geochemistry; S. Ghose, ed.; Springer-Verlag; in press.

Burton, B. P.; Davidson, P. M.; "Order-Disorder in Omphacitic Pyroxenes: A Model for Coupled Substitution in the Point Approximation." Reply American Mineralogist, 73, 916 (1988).

Burton, B. P.; Davidson, P. M.; "Short-Range Order and Frustration in Omphacite." Physics and Chemistry of Materials, 15, 570 (1988).

Burton, B. P.; Kattner, U.; "The Al-Cu-Pb (Aluminum-Copper Lead) System." Bull. Alloy Phase Diagrams; in press.

Coriell, S. R.; McFadden, G. B.; "Double Diffusive Convection during Directional Solidification: Nonlinear Analysis." Proceedings of Tenth U.S. National Congress of Applied Mechanics, J. P. Lamb, ed.; American Society of Mechanical Engineers; New York, NY; 233 (1987).

Davidson, P. M.; Burton, B. P.; "Short-Range Order and Frustration in Omphacite." EOS, 69, No. 16, 521 (1988).

deWit, R.; Low, S. R.; Fields, R. J.; "Ispitivanja Zaustavljanja Prsline u Sirokim Plocama: Razvoj Eksperimentalnih Postupaka." Perspektive Razvoja i Primene Mehanike Loma; S. Sedmak, ed.; Gosa, Belgrade, 201 (1987).

Fong, J. T.; Fields, R. J., editors; "Basic Questions in Fatigue, Volume I," ASTM STP 924 (American Society for Testing and Materials), Philadelphia, 389 (1988).

Handwerker, C. A.; "Diffusion-Induced Grain Boundary Migration." 1989 McGraw-Hill Yearbook of Science and Technology; S. P. Parker, ed.; in press.

Handwerker, C. A.; Cahn, J.; "Microstructural Control Through Diffusion-Induced Grain Boundary Migration." Polysilicon Films and Interfaces; Proceedings of Mat. Res. Soc. Symp., 106, 127 (1988).

Hardy, S. C.; "The Surface Tension of Liquid Silicon." Properties of Silicon, The Institute of Electrical Engineers, 58 (1988).

Hardy, S. C.; Voorhees, P. W.; "Ostwald Ripening in a System with a High Volume Fraction of Coarsening Phase." Met. Trans. (1988) in press.

Kattner, U.; Burton, B. P.; "The Al-Cu-Bi (Aluminum-Copper Bismuth) System." Bull. Alloy Phase Diagrams; in press.

Kikuchi, R.; Burton, B. P.; "Calculation of Some Oxide Systems Using the Cluster Variation Method." Physica B, 150, 132 (1988).

Ledbetter, H. M.; Fields, R. J.; Reno, R. C.; "Texture in Recrystallized Copper Rod: An Ultrasonic-Velocity, Neutron Diffraction Study." Proceedings of the Eighth International Conference on Textures of Materials; Santa Fe (1987); in press.

Lee, D. J.; Vaudin, M. D.; Handwerker, C. A.; Kattner, U. R.; "Phase Stability and Interface Reactions in the Al-SiC System." Proceedings of Mat. Res. Soc. Symp. for High-Temperature, High-Performance Composites; in press.

Liu, T.-S.; Fields, R. J.; Fariborz, S. J.; Harlow, D. G.; Delph, T. J.; "Experimental Observations and Analysis of Creep Cavitation in AISI 304 Type 304 Stainless Steel." Acta Met.; in press (1988).

McFadden, G. B.; Boisvert, R. F.; Coriell, S. R.; "Nonplanar Interface Morphologies during Unidirectional Solidification of a Binary Alloy, II. Three-Dimensional Computations." J. of Crystal Growth, 84, 371 (1987).

McFadden, G. B.; Coriell, S. R.; "Solutal Convection during Directional Solidification." Proceedings of the First National Fluid Dynamics Congress, paper #88-3635; American Institute of Aeronautics and Astronautics, Washington, DC; 1572; 1988.

McFadden, G. B.; Coriell, S. R.; Alexander, J. I. D.; "Hydrodynamic and Free Boundary Instabilities during Crystal Growth: The Effect of a Plane Stagnation Flow." Communications on Pure and Applied Mathematics, 16, 683 (1988).

McFadden, G. B.; Coriell, S. R.; Sekerka, R. F.; "Effect of Surface Tension Anisotropy on Cellular Morphologies." J. Crystal Growth, 91 180 (1988).

Mueller, B. A.; Schaefer, R. J.; Perepezko, J.; "The Solidification of Al-Mn Powders." J. Mater. Res.,2, 809 (1987).

Murray, J. L.; McAlister, A. J.; Schaefer, R. J.; Bendersky, L. A.; Biancaniello, F. S.; Moffat, D. L.; "Stable and Metastable Phase Equilibria in the Al-Mn System." Met. Trans. A, 18A, 385 (1987).

Naus, D. J.; Bass, B. R.; Keeny-Walker, J.; Fields, R. J.; deWit, R.; Low, S. R., III; "Summary of HSST Wide Plate Crack Arrest Tests and Analysis." Proceedings of the U.S. Nuclear Regulatory Commission Fifteenth Water Reactor Safety Information Meeting, NUREG/CP-0091; Vol. 2; A. J. Weiss, ed.; Office of Nuclear Regulatory Research, USNRC; Washington, DC (1988).

Pugh, C. E.; Naus, D. J.; Bass, B. R.; Nanstad, R. K.; deWit, R.; Fields, R. J.; Low, S. R., III; "Crack Arrest Behavior in Wide Single Edge Notched Plates of a Light Water Reactor Pressure Vessel." Trans. of 9th International Conf. on Solid Mechanics in Reactor Technology; Lausanne, Switzerland; August 17-21; paper G1-4 (1987).

Pugh, C. E.; Naus, D. J.; Bass, B. R.; Nanstad, R. K.; deWit, R.; Fields, R. J.; Low, S. R., III; "Wide Plate Crack Arrest Tests Utilizing a Prototypical Pressure Vessel Steel." Int. J. of Pressure Vessels and Piping, 31 (1988).

Reno, R. C.; Clark, A. V.; Blessing, G. V.; Fields, R. J.; Govada, A.; Thomson, R. B.; Del Santo, P. P.; Mignogna, R. B.; Smith, J. F.; "Use of Neutron Pole Figures to Calibrate Ultrasonic Techniques for On-line Texture Control of Aluminum Plates." Intelligent Processing of Materials and Advanced Sensors; H. N. G. Wadley, P. A. Parrish, B. B. Rath, S. M. Wolf; eds.; The Metallurgical Society; Warrendale, PA; 77 (1987).

Reno, R. C.; Fields, R. J.; Clark, A. V., Jr.; "Crystallographic Texture in Rolled Aluminum Plates: Neutron Pole Figure Measurements." Review of Progress in Quantitative Nondestructive Evaluation; D. O. Thompson, D. E. Cimenti, eds.; Plenum, New York, NY; 1439 (1988).

Rhee, W.-H.; Handwerker, C. A.; Yoon, D.; "The Coherency Strain Induced Instability of Solid-Liquid Interfaces in the Mo-Ni System." Proceedings of Mat. Res. Soc. Symp.; Interfacial Structure, Properties, Design; Vol. 122; in press.

Schaefer, R. J.; "Phase Selection in Surface Resolidification." In Rapid Solidification Processing: Principles and Technologies IV; R. Mehrabian, ed.; 249 (1988).

Schaefer, R. J.; Bendersky, L. A.; "Metallurgy of Quasicrystals." Chapter 3 in Aperiodic Crystals; M. V. Jaric, ed.; Academic Press; (1988).

Sekerka, R. F.; Voorhees, P. W.; Coriell, S. R.; McFadden, G. B.; "Initial Conditions Implied by $t^{1/2}$ Solidification of a Sphere with Capillarity and Interfacial Kinetics." J. Crystal Growth, 87, 415 (1988).

Sims, J. S.; Redmiles, D. F.; Clark, J. B.; "ASM/NBS Numerical and Graphical Database for Binary Alloy Phase Diagrams" in Metallurgical Data Bases, (American Society for Metals, Metals Park) in press.

Stauss, G. H.; Rubinstein, M.; Frieble, E. J.; Bennett, L. H.; Schaefer, R. J.; "Nuclear-Magnetic-Resonance Powder Patterns in Al_6Mn, Al_4Mn, and $Al_{12}Mn$ Polycrystals." Phys. Rev. B, 35, 2700 (1987).

Vandiver, P.; Cort, L.; Handwerker, C. A.; "Variations in the Practice of Ceramic Technology in Different Cultures; A Comparison of Korean and Chinese Celadon Glazes." Advances in Ceramics; in press.

Wheeler, A. A.; Coriell. S. R.; McFadden, G. B.; Hurle, D. T. J.; "The Effect of an Electric Field on the Morphological Stability of the Crystal-Melt Interface of a Binary Alloy." J. Crystal Growth, 88, 1 (1988).

Advanced Sensing Group

Blendell, J. E.; Chiang, Cranmer, D. C.; Freiman, S. W.; Fuller, Jr., E. R.; Drescher-Krasicka, E.; Johnson, Ward L.; Ledbetter, H. M.; Bennett, L. H.; Swartzendruber, L. J.; Maronenko, R. B.; Mykleburst, R. L.; Bright, D. S.; and Newbury, D. E.; "Relationship of Electrical, Magnetic, and Mechanical Properties to Processing in High-Temperature Superconductors in Chemistry of High-Temperature Superconductors." American Society Symposium, Series No. 351, 240 (ACS, Washington, D. C.) (1987).

Johnson, W. L.; Drescher-Krasicka, E.; Blendell, J. E.; Blendell, J. E.; Chiang, C. K.; and Wadley, H. N. G.; "Ultrasonic Velocity and Attenuation in $Y_1Ba_2C_{u3}O_{7-x}$." Phys. Rev. Let.; in press.

Kahn, A. H.; Mester, M. L.; "An Eddy Current Sensor for the Measurement of Resistivity and Temperature of Aluminum Rod During Extrusion Processing." Review of Progress in Quantitive NDE, 6; in press (1988).

Kahn, A. H.; Mester, M. L.; and H. N. G. Wadley; "Measuring Internal Temperatures of Aluminum Extrusion." Proc. 4th Int. Aluminum Extrusion Technology Seminar, Apr., 1988, Chicago, Ill.

Kahn, A. H.; Lowney, J. R.; "Valence Band Effective Masses of GaAs," J. Appl. Phys., 64, 447 (1988).

Kahn, A. H.; Danyluk, S.; Lee, S. W.; and Ahn, J. H.; "Resistivity of Linear Scratches in Doped (100) n-type Single Crystal Silicon." J. Appl. Phys. 63 (9), May 1, 1988.

Kahn, A. H.; "Boundary Integral Equation Methods for Two Dimensional Models of Crack Field Interactions." presented at The Technical Cooperative Program Workshop, Dayton, Ohio, August 1987, (to be published in J. Nondestr. Eval., (1988-1989).

Norton, S. J.; "Computing Ray Trajectories Between Two Points: A Solution to the Ray Linking Problem." J. Opt. Soc. Am. A 4, 1919 (1987).

Norton, S. J.; "Mossbauer Imaging." Nature 330, 151 (12-18 Nov. (1987).

Norton, S. J.; Atzmony, U.; Swartzendruber, L. J.; Bennett, L. H.; "Mossbauer Imaging: Experimental Result." Nature 330, 153 (12-18 Nov. 1987).

Norton, S. J.; "Mossbauer Imaging." NBS J. Research 92, 325 (1987).

Norton, S. J.; Simmons, J. A.; Kahn, A. H.; and Wadley, H. N. G.; "Research on Inverse Problems in Materials Science and Engineering." Proceedings of the NATO Advanced Research Workshop on Signal Processing and Pattern Recognition in Nondestructive Evaluation of Materials, 1987, Springer-Verlag.

Norton, S. J.; "Mossbauer Imaging." Physics Bulletin 39, No. 5 (1988).

Norton, S. J.; "Iterative Seismic Inversion." Geophys. J. Roy. Astr. Soc.; in press.

Norton, S. J.; "Tomographic Reconstruction of Two-Dimensional Vector Fields: Application to Flow Imaging." Geoph. J. Roy. Astr. Soc.; in press.

Simmons, J. A.; Drescher-Krasicka, E.; Wadley, H. N. G.; Rosen, M.; and Hsieh, T. M.; "Ultrasonic Methods for Characterizing the Interface in Composites." Review of Progress in QNDE, Vol. 7, Ed. D. O. Thompson and D. E. Chimenti; in press.

Wadley, H. N. G.; Kahn, A. H.; Gefen, Y.; and Mester, M. L.; "Eddy Current Measurement of Density During Hot Isostatic Pressing." Review of Progress in QNDE, Col. 7, Eds. D. O. Thompson and D. E. Chimenti.

Wadley, H. N. G.; Simmons, J. A.; Drescher-Krasicka, E.; Rosen, M.; Clough, R. B.; Hsieh, T.; Hirschman, K.; and Biancaniello, F. S.; "Composite Materials Interface Characterization." NBSIR 87-3630, March 1988.

Wadley, H. N. G.; Simmons, J. A.; and Drescher-Krasicka, E.; "Ultrasonic Propagation at Cylindrical Metal-Ceramic Interfaces in Composites." MRS Symposium High Temperature Composites, Ed. F. Lemkey, S. Fishman and A. Evans, Reno, Nevada; in press (1988).

Wadley, H. N. G.; Biancaniello, F. S.; and Clough, R. B.; "Micromechanical Studies of Model Metal Matrix Composites." Proc. MRS. Symposium, High Temperature Composites, Ed. F. Lemkey, S. Fishman and A. Evans, Reno, Nevada; in press (1988).

Wadley, H. N. G.; Kahn, A. H.; and Johnson, W.; "Advanced Sensing of Materials Processing." MRS Symposium Proceedings, Process Diagnostics; Materials, Combustion, Fusion, Ed. A. K. Hays, A. C. Eckbreth, G. A. Campbell, Reno, Nevada, 117, 109 (1988).

Wadley, H. N. G.; and Rogers, C. D.; "Internal Discontinuity Sensor Needs for Steel: Report of Workshop." NBSIR 88-3731, June 1988.

Patent:

Wadley, H. N. G.; "Method of Determining Subsurface Property Value Gradient." Patent Number: 4,765,750, August 23, 1988.

Corrosion Group

Anderson, D.B.; Laverty, G.J.; "Corrosion Data for Materials Performance Characterization." presented at First International Symposium on Computerization and Networking of Materials Property Databases, Philadelphia, November 1987, to be published in ASTM Special Technical Publication.

Anderson, D.B.; "Standards for Corrosion Database Development." NACE Corrosion/88, Paper #119 (1988).

Bertocci, U.; "Modeling of Crack Chemistry in Cu-Au Alloys." J. Electrochemical Society; in press.

Bertocci, U.; "Statistics of Localized Breakdown Noise." NACE Sympoium on Localized Corrosion; in press.

Bertocci, U.; Pugh, E.N.; "Modeling of Electrochemical Processes During Transgranualr Stress Corrosion Cracking of Copper Base Alloys." Proc. International Conference on Metallic Corrosion (1987).

Early, J.E.; Interrante, C.G.; Low, S.R.; Fields, R.J.; "Dynamic Mechanical Properties of two AAR-M128 and One ASTM-A212-B Tank Car Head Plates." NBSIR 88-3690, (1988).

Escalante, E.; "Concepts of Underground Corrosion." ASTM STP 1013 on The Effects of Soil Characteristics on Corrosion, May 1987; in press.

Escalante, E.; "Elimination of iR Error in Measurements of Corrosion in Concrete." ASTM STP on Ohmic Electrolyte Resistance Measurement and Compensation; May 1988; in press.

Escalante, E.; "Measuring the Rate of Corrosion of Steel in Concrete." ASTM STP on Corrosion Rates of Steel in Concrete; June 1988; in press.

Fraker, A.C.; Sung P.; "Corrosion and Degradation of a Polyurethane/Co-Ni-Cr-Mo Pacemaker Lead." J. Biomed. Mater. Res.: Applied Biomaterials, Vol. 21 No. A3, 287 (1987).

Fraker, A.C.; "Corrosion of Zircaloy, Report to the U. S. Nuclear Regulatory Commission." Washington, DC; February (1988).

Fraker, A.C.; Harris, J.S.; "Corrosion Behavior of Low Carbon Steel in High pH Aqueous Media." Report to the U. S. Nuclear Regulatory Commission, Washington, DC; March (1988).

Hall, D.E.; "Sintered Lead Negative Plates Made with Leachable Pore Formers." Electrochemical Society Extended Abstracts, 87-2 (1987).

Hall, D.E.; Sarver, J.M.; Gothard, D.O.; "AB_5-Catalyzed Hydrogen Evolution Cathodes." Proc. Symp. Electochem. Eng. in the Chlor-Alkali and Chlorate Industries, The Electrochemical Society, Inc. (1987).

Hall, D.E.; Stafford, G.R.; "The Corrosion Behavior of Electrodeposited Aluminum-Manganese Alloys." Electrochemical Society Extended Abstracts, 87-2 (1987).

Interrante, C.G.; Escalante, E.; Fraker, A.C.; Ondik, H.; Plante, E.; Ricker, R.E.; Ruspi, J.; "Evaluation and Compilation of DOE Waste Package Test Data." Biannual Report, August 1987 - January 1988, NUREG/CR-4735, 4 (1988).

Interrante, C.G. (editor); Escalante, E; Fraker, A.C.; Hall, D.E.; Harrison, S.; Liggett, W.; Linzer, M.; Ricker, R.; Ruspi, J.; Shull, R.; "Evaluation and Compilation of DOE Waste Package Test Data." Biannual Report, February 1987 - July 1987." NUREG/CR-4735, 3 (1988).

Interrante, C.G. (editor); "Evaluation and Compilation of DOE Waste Package Test Data: Biannual Report, August 1987 - January 1988." NUREG/CR-4735, 4, (1988).

Ricker, R.E.; Duquette, D.J.; "The Role of Hydrogen in Corrosion Fatigue of High Purity Al-Zn-Mg Exposed to Water Vapor." Metall. Trans. A., V19A, 1775 (1988).

Trim, J.D.; Anderson, D.B.; Laverty, G.J.; "The NACE-NBS Corrosion Data Program." Proc. 10th International Congress on Metallic Corrosion (1987).

Electrodeposition Group

Goldman, A. I.; Long, G. G.; Bennett, L. H.; Lashmore, D. S.; Kuriyama, M.; "Observations of Two Structurally Distinct States in Glassy Ni-P Using EXAFS." J. Electrochem. Soc. 135, 9, (1988).

Grushko, B.; Stafford, G. R.; "The Effect of Mn Content on the Microstructure of Al-Mn Alloys Electrodeposited at 150 °C." Met Trans.; in press.

Lashmore, D. S.; "Annual Review of Materials Science-Invited to Prepare a Review of the Pulsed Electrochemical Deposition of Alloys"; in press.

Lashmore, D. S.; Dariel, M. P.; "Electrodeposited Cu-Ni Textured Superlattices." J. of the Electrochem. Soc., 135, 5, 1218 (1988).

Ogburn, F.; Brown, H. J.; "Aging Effects on XRF Measurements of Solder Coatings." J. of the American Electroplaters and Surface Finishers Soc.; in press.

Stafford, G. R.; "The Electrodeposition of an Aluminum-Manganese Metallic Glass from Molten Salts." Proc. Corr. Electrochem. and Cat. of Metallic Glasses, Vol.88-1, 172 Meet. Electrochem. Soc., Honolulu, Hawaii (1987).

Stafford, G. R.; "The Electrodeposition of an Aluminum-Manganese Metallic Glass from Molten Salts." J. Electrochem. Soc.; in press.

Wheeler, N. S.; "The Effect of Magnesium Complex Ions on the Corrosion Behavior of Magnesium." Ph.D. Thesis, American University, Dept. of Chemistry, April 5, 1988.

Magnetic Materials

Adrian, F. J., Bohandy, J., Kim, B. F., Moorjani, K., Wallace, J.S., Shull, R. D., Swartzendruber, L. J., Bennett, L. H., "Magnetic Field Dependence of the Superconductivity in Bi-Sr-Ca-Cu-O Superconductors". Physica C, 156, 184 (1988).

Atzmony, U., Swartzendruber, L. J., Bennett, L. H., Dariel, M. P., Lashmore, D. S., Rubinstein, M., Lubitz, P., "Magnetication and Magnetic Aftereffect in Textured Ni/Cu Compositionally-modulated alloys. J. Mag. Magn. Matls., 69, 237 (1987).

Atzmony, U., Shull, R. D., Chiang, C. K., Swartzendruber, L. J. and Bennett, L. H., "Constricted Diamagnetic Hysteresis Loops Observed for the High T_c Superconductors". J. Appl. Phys. 63, 4179 (1988).

Atzmony, U., Swartzendruber, L. J. and Bennett, L. H., "Temperature Hysteresis and Time Dependent Effect in the Initial Susceptibility of Rapidly Solidified Monel". Scripta Metall. 22, 721 (1988).

Bennett, L. H., Watson, R. E., Melamud, M, "The Roles of Atomic Volume and Disclinations in the Magnetism of the Rare Earth-3D Hard Magnets". J. de Physique; in press (1988).

Bohandy, J., Adrian, F. J., Kim, B. F., Moorjani, K., Shull, R.D., Swartzendruber, L. J., Bennett, L. H., Wallace, J.S., "Magnetic-field-modulated Microwave-absorption Detection in a Bi-Sr-Ca-Cu-O Superconductor". J. of Superconductivity; in press (1988).

Goldman, A. J., Long, G. G., Bennett, L. H., Lashmore, D. S. ,and Kuriyama, M., "Observation of Two Structurally Distinct States in Ni-P Glasses Using EXAFS". J. of Electrochemical Society, 135, 8 (1988).

Huang, C. Y., Shapira, Y., McNiff, E. J., Peters, P. N., Schwartz, B. B., Wu, M. K., Shull, R. D., "Magnetic Hysteresis of High-Temperature $YBa_2Cu_3O_x$-AgO Superconductors: Explanation of Magnetic Suspension". Modern Physics Letter B; in press (1988).

Jones, S. A., Shull, R.D., McAlister, A. J., Kaufman, M. J., "Microstructural Studies of Ti-Al Alloys in the Vicinity of the Eutectoid Reaction". Scripta Metallurgica, 22, 1235 (1988).

Kim, B. F., Phillips, T. E., Green, W. J., Swartzendruber, L. J., Shull, R. D., Bennett, L. H., Wallace, J. S., "Superconducting Thin Films of Bi-Sr-Ca-Cu-O Obtained by Laser Ablation Processing". Appl. Phys. Letter; in press (1988).

Lundy, D., Ritter, J., Swartzendruber, L., Shull, R. D., Bennett, L. H., "Studies of Magnetic Flux Penetration in a Chemically Synthesized Bi(Pb)SrCaCuO Superconductor". Physica C; in press (1988).

Melmed, A. J. Shull, R. D., Chiang, C. K. and Fowler, H. A., "Field Ion Microscopy of the High T_c Superconductor $Ba_2YCu_3O_{7-x}$". J. Appl. Phys., 63, 4232 (1988).

Melmed, A. J., Shull, R. D., Chiang, C. K., and Fowler, H. A., "Possible Evidence for Superconducting Layers in Single Crystal $YBa_2Cu_3O_{7-x}$ by Field Ion Microscopy". Science, 239, 176 (1988).

Melmed, A. J., Shull, R. D., Chiang, C. K., and Fowler, H.A., "The Atomic Fingerprint of $YBa_2Cu_3O_{7-x}$ - type High-Temperature Superconductors Observed by Field Ion Microscopy". Mat. Sci. and Eng., 100, L27 (1988).

Melmed, A. J., Shull, R. D. and Chiang, C. K., "Progress in Field Ion Microscopy Imaging of High-T_c Superconducting Oxides". J. de Physique; in press (1988).

Melamud, M., Watson, R. E., and Bennett, L. H., "A Wigner-Seitz Local-Environment Study of the High T_c Superconductors". Phys. Rev. B., 38, 4 (1988).

Moorjani, K. Bohandy, J. Adrian, F. J. Kim, B. F., Atzmony, U. Shull, R. D. Chiang, C. K. Swartzendruber, L. J. and Bennett, L. H., "Magnetic Behavior of Both Superconducting Thin Films and Their Deposition Targets". J. Appl. Phys., 63, 4199 (1988).

Shull, R. D., Atzmony, U., Shapiro, A. J. Swartzendruber, L. J., Green, W. L. and Moorjani, K., "Magnetic Properties of Thin Film Ag+Iron Oxide Granular Metals". J. Appl. Phys., 63, 4261 (1988).

Swartzendruber, L. J., "Quantitative Problems in Magnetic Particle Inspection". QNDE Conf. Proceedings; in press (1988).

Swartzendruber, L. J., Bennett, L. H., "Mössbauer Hyperfine Fields in $RBa_2(Cu_{0.97}Fe_{0.03})_3O_{7-x}$ Superconductors". J. de Physique; in press (1988).

Swartzendruber, L. J., Bennett, L. H., "Mössbauer Study of the Effect of Oxygen Stoichiometry on the High T_C Superconductor $Y_1Ba_2(Cu_{0.97}Fe_{0.03})_3O_{7-x}$". J. of Superconductivity; in press (1988).

Wallace, J. S., Ritter, J. J., Fuller, E., Bennett, L. H., Shull, R.D. and Swartzendruber, L. J., "Synthesis & Magnetic Properties of the $BiSrCaCu_2O_x$ 80 and 110 K Superconductors"; in press (1988).

Watson, R. E., Bennett, L. H., and Melamud, M., "Disclinations: The Magnetism and the Magnetic Anisotropies of the Rare Earth-3d Transition Metal Hard Magnets". J. Appl. Phys., 63, 3136-3138 (1988).

Wong-Ng, W., Cook, L. P, Chiang, C. K., Swartzendruber, Bennett, L. H., Blendell, J., and Minor, D., "Structural Phase Transition Study of $Ba_2YCu_3O_{7-x}$"; in press (1988).

High Temperature Chemistry

Cook, L. C.; Plante, E. R.; Bonnell, D. W.; Hastie, J. W.; "High Temperature Chemistry of Stored Chemical Energy Systems for Propulsion." NBSIR-87-3601 (1987).

Hastie, J. W.; Bonnell, D. W.; Plante, E. R.; "Experimental and Model Determinations of Coal Mineral and Slag Phase Equilibria." Proc. Symp. High Temperature Materials Chemistry IV, The Electro-chemical Society, Pennington, N. J. (1987).

Hastie, J. W.; Schenck, P. K.; Bonnell, D. W.; "Ceramic Thermochemistry and Kinetics from Laser-Induced Vaporization Mass Spectrometry." Proc. Symp. High Temperature Materials Chemistry IV, The Electro-chemical Society, Pennington, N. J. (1987).

Bonnell, D. W.; Schenck, P. K.; Hastie, J. W.; "Laser Induced Vaporization Time Resolved Mass Spectrometry of Refractories." Proc. Int. Symp. Laser Processes for Microelectronic Applications, The Electrochemical Society,Pennington, N. J. (1987).

Hastie, J. W.; "Measurement Advances and Limitations in High Temperature Mass Spectrometry." Proc. Fourth National Symposium Mass Spectrometry, India Institute of Science, Bangalore, India (1988).

Plante, E. R.; Bonnell, D. W.; Hastie, J. W.; "Experimental and Theoretical Determination of Oxide Glass Vapor Pressures and Activities." Proc. Int. Conf. Advances in the Fusion of Glass, Alfred, NY, American Ceramic Society; in press, (1988).

Hastie, J. W.; Bonnell, D. W.; Plante, E. R.; "Experimental and Model Determination of Coal Mineral and Slag Phase Equilibria and Alkali Vapor Transport, VIII." Proc. Eng. Foundation Conf. "Mineral Matter and Ash Deposition from Coal," February 21-26, Santa Barbara, CA; in press (1988).

Hastie, J. W.; Bonnell, D. W.; "Thermodynamic Activity Predictions for Molten Slags and Salts, IX." Proc. 3rd Int. Conf. Molten Slags and Fluxes, June 27-29, Glasgow, UK (Inst. Metal); in press (1988).

Hastie, J. W.; "Coupling Mass Spectrometry with High Pressure/High Temperature Systems." Advances in Mass Spectrometry (Heyden and Sons, Ltd.) Proc. 11th Int. Mass Spec. Conf., Bordeaux, August 29 - September 2; in press (1988).

Schenck, P. K.; Bonnell, D. W.; Hastie, J. W.; "Laser Vaporization Mass Spectrometry of Refractory Materials: Graphite and $YBa_2Cu_3O_x$." Journal of the American Vacuum Society Proc. 1988 Joint Int. Laser Science and American Vacuum Soc. Conf.; in press.

Hastie, J. W.; Bonnell, D. W.; Plante, E. R.; "Experimental Determination and Model Prediction of Glass Thermochemistry X." Proc. XV Int. Congress on Glass, Leningrad, USSR, July 2-7; in press (1989).

Schenck, P. K.; Dennis, J. R. (Acers); "PC-Access to Ceramic Phase Diagrams." Proc. First Int. Symp. on Computerization and Networking of Materials Property Databases (SLP1017) ASTM (1988).

Mechanical Properties of Metals

Finn, R. D.; Shives, T. R.; Brady, C. H.' Plascjak, P.; Meyer, W.; Sheh, Y.; Larson, S.; "Correlation of Hardness with Radiation Induced Defects in Metal Foil Windows." Proceedings - Seventh International Symposium on Radiopharmaceutical Chemistry, Groningen, The Netherlands; in press (1988).

Finn, R. D.; Shives, T. R.; Brady, C. H.; Plascjak, P.; Meywer, W.; Sheh, Y.; "Microhardness Evaluations of Cycletron Fail Windows." Proceedings - Tenth Conference on the Application of Accelerators in Research and Industry; in press (1988).

Gross, J. L.; Yokel, F. Y.; Wright, R. G.; Fanney, A. H.; Smith, J. H.; Hicho, G. E.; Shives, T. R.; "Investigation into Ashland Oil Storage Tank Collapse on January 2, 1988." NBSIR 88-3792 (1988).

Gross, J. L.; Smith, J. H.; Wright, R. U.; "Ashland Tank Collapse Investigation." Journal of Performance of Constructed Facilities; in press (1988).

Hicho, G. E.; Fields, R. J.; "Effects of Varying the Austenitizing Temperature and Cooling Rate on the Ability of HSLA-80 to Achieve a Yield Strength Comparable to HSLA-80." Journal of Heat Treating; in press (1988).

Hicho, G. E.; Smith, L. C.; "Superheater Sample Taken from Potomac River Unit 4." Letter Report to PEPCO (October 7, 1987).

Hicho, G. E.; Smith, L. C.; "Waterwall Tube Sample Removed from Chalk Point Unit 2." Letter Report to PEPCO (October 10, 1987).

Hicho, G. E.; Smith, L. C.; "First Stage, Second Pass Reheater Outlet Header Stub Sample Taken from Chalk Point Unit 1." Letter Report to PEPCO (November 23, 1987).

Hicho, G. E.; Smith, L. C.; "Waterwall Tube Failure, Chalk Point Unit 3." Letter Report to PEPCO (March 7, 1988).

Hicho, G. E.; Smith, L. C.; "Superheater Tube Failure, Potomac River Unit 3. Letter Report to PEPCO (March 7, 1988).

Hicho, G. E.; Smith, L. C.; "Waterwall Tube Sample, Chalk Point Unit 1." Letter Report to PEPCO (July 15, 1988).

Hicho, G. E.; Smith, L. C.; "Chromized Tubes, Morgantown Unit 2." Letter Report to PEPCO (July 21, 1988).

Hicho, G. E.; Smith, L. C.; "Babcock and Wilcox Chromized Tubes." Letter Report to PEPCO (August 1, 1988).

Hicho, G. E.; Smith, L. C.; "Chromized Tube Removed from Waterwall Panel in Service in Morgantown Unit 2 since November 1981." Letter Report to PEPCO (August 3, 1988).

Hicho, G. E.; Smith, L. C.; "Waterwall Tube Samples, Chalk Point Unit 3." Letter Report to PEPCO (August 15, 1988).

Hicho, G. E.; Smith, L. C.; "Benning Road Unit 3 Turbine Bolt Failure." Letter Report to PEPCO (August 8, 1988).

Huang, Y.; Hicho, G. E.; Fields, R. J.; "J-Integral, R-Curve Testing of 5052 H112 Aluminum Utilizing DC Potential Drop and Infrared Thermographic Methods." Proceedings of the Seventh International Conference on Fracture, Houston, Texas, March 1989.

Huang, Y.; Hicho, G. E.; Fields, R. J.; "Infrared Measurements of Heating and Cooling Emissions in Aluminum and Steel During Tensile and Cyclic Loading" NBSIR 87-3653, February 1988.

Kreider, K.; Shapiro, A. J.; "Sputtered Thin Film Y Ba_z Cu_3O_2." Proceedings - American Vacuum Society Symposium on High T_C Super Conducting Thin Films, Oct. 3-7; in press (1988).

McHenry, H. I.; Purtscher, P. T.; Shives, T. R.; "Observations of Hydrogen Damage in a Failed Pressure Vessel." Corrosion Science, 27, No. 10/11, 1041 (1987).

Shives, T. R.; "Microhardness and Thickness Measurements on Bullets." memo report to NIST Law Enforcement Standards Laboratory (Jan. 1988).

Shives, T. R; Smith, L. C.; "Tensile Testing of Vehicle Restrain Devices." Letter report to the US Secret Service (Dec. 1987).

Shives, T. R.; Hardness Measurement on the Lead Core of Two Bullets." memo report to NIST Law Inforcement Standards Laboratory (Feb. 1988).

Shives, T. R.; "Interim report on the Results of NBS tests on Aramid Fiber Rope." Letter report to the US Coast Guard (Feb. 1988).

Shives, T. R.; "Hardness and Thickness Measurements on Bullets." memo report to NIST Law Enforcement Standards Laboratory (Sept. 1988).

Shives, T. R.; Smith, L. C.; "Testing of School Bus Flooring." memo report to the National Transportation Safety Board (Oct. 1988).

Shives, T. R.; Mertaugh, L. J.; "Detection, Diagnosis and Prognosis of Rotating Machinery to Improve Reliability, Maintainability, and Readiness through Application of New and Innovative Techniques." Proceedings of the 41st Meeting of the Mechanical Failures Prevention Group; in press (1988).

Shull, R. D.; Shapiro, A. J.; "Magnetic Properties of Thin Film Silver-Iron Oxide Granular Metals." J. Appl. Phys. 63 C81, 4261 (1988).

Shull, R. D.; Ritter, J. J.; Swartzenduber, L. J.; Bennett, L. H.; "Magnetic Properties of Iron-Silica GEL Nanocomposites." Proceedings - Symposium on Multicomponent Ultrasonic Microstructures Materials Research Society, Boston; in press (1988).

Siewert, T.; Hicho, G. E.; "Choosing a Welding Procedure to Produce a 10 Meter Long Tensile Specimen." Welding Journal, Feb. 1988, 35

Wright, R. N.; Smith, J. H.; "Oil Storage Tank Collapse of Ashland - Floreffe Terminal." Materials Evaluation, April 1988, 508.

INDUSTRIAL AND ACADEMIC INTERACTIONS

The research programs of the Metallurgy Division are designed and carried out in support of industrial and scientific needs. Specialized facilities within the Division, including metals processing and nondestructive evaluation, attract scientists from both academic and industrial organizations for cooperative research efforts. Interactions with industry, universities, and professional organizations are viewed as an important element of our work with collaborative programs, consulting and general involvement with outside groups being a long standing practice. For example, the Metallurgy Division has been working for more than 70 years with the steel industry to improve the durability and performance of alloys.

In 1988, the Division performed collaborative research with many private organizations through its Research Associate and Guest Scientist programs and other arrangements. Representative examples of such interactions include:

INDUSTRY

1. ACerS (American Ceramic Society)

   Dr. Peter Schenck is collaborating with ACerS and the industrial sponsors of the NBS-ACerS phase diagram optimization program, in the development of a graphical phase diagram database for ceramic and other inorganic systems. During the past year a prototype PC version was released to ACerS and its industrial sponsors.

2. ALCOA

   A cooperative program with the Alloy Technology Division of ALCOA (Dr. A.K. Vasudevan) and the Corrosion group is in progress. This program focuses on the influence of grain boundary precipitates on the susceptibility of Al-Li and Al-Li-Cu alloys to intergranular stress corrosion cracking. Samples with differing precipitate size distributions are prepared at ALCOA and tested at NIST.

3. Aluminum Association

   The first phase of the cooperative project of NIST and the Aluminum Association has been brought to a successful conclusion with the execution of a plant demonstration of the eddy current sensor developed for measuring temperature of extruded aluminum products during processing. The Aluminum Association and NIST have agreed to continue the project for further two years with the objectives of measuring temperatures in more complex shapes and of obtaining information on the non-uniformity of temperature profiles.
   Mr. Michael L. Mester will continue as Research Associate for the Aluminum Association.

4. American Association of Railroads (AAR)

The Railway Tank Car Safety Research and Test Committee of the AAR Research Progress Institute has established a collaborative research with the NIST program funded by the Federal Railroad Administration (FRA) to conduct research on the properties of tank car materials.

5. American Cyanamid Corporation

A joint program between the Electrodeposition Group and American Cyanamid Corporation is addressing the problem of interfacial interdiffusion of carbon and nickel at elevated temperatures. The research is being done by a Research Associate (N. Wheeler) in collaboration with members of the Electrodeposition Group (D. S. Lashmore and C. E. Johnson), with others in the Metallurgy Division (A. Shapiro, C. Handwerker, U. Kattner), and with the Center for Analytical Chemistry (K. Pratt).

6. American Iron and Steel Institute

The Advanced Sensing Group has continued a strong collaboration with the American Iron and Steel Institute (AISI) during FY 88. The interaction began in 1983 with the signing of a memorandum of understanding and agreement to research and develop ultrasonic approaches for internal temperature distribution and pipe/porosity sensors for control of steel processing. The work on the pipe/porosity was completed with the successful evaluation of a prototype system in FY 86. The sensor was the recipient of an IR-100 award in FY 87 and is now in routine use in at least one steel mill. Research continues with the temperature sensor toward extending interface imaging methods for solidifying bodies to alloys with extensive regions of liquid-solid coexistence.

7. Artech Corporation

Electrochemical measurements were conducted as part of a study to develop a standard test for determining surface condition of metal dental implants. One of the products of the Artech Corporation is dental implants.

8. ASM INTERNATIONAL (American Society for Metals)

The technical activities of the joint NIST/American Society for Metals Data Program for alloy phase diagrams are centered at NIST. The editor and associate editor of the Bulletin of Alloy Phase Diagrams are J. B. Clark and B. Burton (both at NIST).

9. BDM Corporation

NIST researchers are collaborating with computer scientists at BDM on a DARPA funded program to develop an Intelligent Processing of Materials Concept for the Hot Isostatic Pressing of Intermetallic Composites.

10. Bethlehem Steel Corporation

Assisted in the evaluation of the Chickasawbogue Bridge failure in Mobile, AL which collapsed because of corrosion of the supporting steel piles immersed in aggressive water.

11. Collaborative Testing Services, Inc.

R. J. Fields collaborated with Charles Leete and Sarah Weitzel of Collaborative Testing Services (CTS) in the selection of materials and specimen fabrication procedures for the interlaboratory comparison testing. The areas in which laboratory comparisons were sought were tensile testing and hardness testing of metals. A method was also jointly devised to handle gradients of materials properties, as naturally occur in long bars, in the evaluation and comparison process.

12. Crucible Materials Corp., General Electric Co., and Hoeganaes Corp.

An industry-NIST consortium has been formed to conduct research on automated measurement and control of powder particle size distributions produced by atomization. Scientists from Crucible Materials, General Electric, and Hoeganaes, which all are companies strongly interested in control of fine rapidly-solidified alloy powders, are collaborating in this consortium work with NIST scientists. The NIST supersonic inert gas metal atomizer (SIGMA) in the Metallurgy Division's metals processing laboratory plays a central role in this work. During the past year the SIGMA has been instrumented to allow real-time measurements by laser diffraction techniques of powder size distributions while they are being produced. Methods of measuring and controlling the liquid droplet formation process are being developed to allow feedback and automated control of atomization processes.

13. DuPont (Wilmington, DE)

Dr. John Hastie is collaborating with Dr. U. Klabunde and others of DuPont on the development of process mechanisms for titanium extractive metallurgy.

14. FIBA, Inc. and Union Carbide Corporation

A collaborative effort is underway between FIBA, Inc. (P. Horrigan), Union Carbide Corporation (R. Tripolet), and NIST (J. H. Smith) to evaluate use of acoustic emission techniques for use in the periodic inspection of large steel pressure vessels. NIST is in the process of developing specific procedures and test criteria to permit the use of acoustic emission techniques for this application.

15. General Electric Co.

The Advanced Sensing Group is collaborating with the Aircraft Engines Division of G. E. to explore potential methods for microstructure

characterization during processing, for sensing the liquid-solid interface during "skull" melting of superalloys and applying acoustic emission techniques to detect residual stress induced microcracking in intermetallic composites.

16. General Electric Co.

A cooperative investigation on the resistance of NIST (D. S. Lashmore, D. R. Kelley and C. E. Johnson) coated fibers to high temperature degradation is underway.

17. General Electric Co.

In an innovative study, the Metallurgical Processing Group and General Electric Co. (S. Miller) performed a cooperative project to apply holographic techniques to visualize the break-up of molten alloys into fine droplets in the NIST high pressure atomization system. This was the first application of holography to atomization processes involving incandescent metals. The extremely short 20 ns laser pulses used in this work provided stop-action pictures never before obtained of this dynamic atomization process.

18. Hoeganaes Corporation

Measurements of the surface tensions of molten steel, its dependence on temperature, and how these vary as a function of alloying content were performed by a guest scientist from Hoeganaes Corp. in collaboration with S. C. Hardy of the NIST Metallurgical Processing Group. These surface tensions are important in controlling liquid droplet formation during production of steel powder by atomization processes.

19. IBM Corporation

An investigation of the magnetic properties of electrochemically produced superlattices is underway under IBM sponsorship. (L. H. Bennett, D. S. Lashmore and R. R. Oberle)

20. Inland Steel

Drs. Plante and Bonnell have been collaborating with Dr. H. Pielet of Inland on the thermochemistry of blast furnace and steel slags and inorganic inclusions in steel. Dr. Pielet provides slag samples and data from plant experience, Dr. Plante is providing thermochemical data from high temperature mass spectrometry, while Dr. Bonnell supplies database and model techniques.

21. Luxfer USA, Inc.

A collaborative effort between NIST (J. H. Smith) and Luxfer USA, Inc. (G. Waite) is ongoing to determine the extent of cracking in seamless aluminum compressed gas cylinders and to develop a reliable test method for inspecting the cylinders in service.

22. Materials Technology Institute of the Chemical Process Industries

MTI, in conjunction with the NACE-NIST Corrosion Data Center (D.B. Anderson), has initiated a project to develop expert systems for selection of materials for storage and handling of hazardous chemicals. Systems are based on rules defined during discussions with consultant experts representing a broad range of industrial experience and are structured to meet the needs of materials specialists involved in design, maintenance and expert assessments. MTI has assigned a Research Associate (C. P. Sturrock) to work in the Data Center as program leader with a goal to develop a minimum of three modules over a thirty month period.

23. Martin Marietta Corporation

Ward L. Johnson and Haydn N. G. Wadley are collaborating with John Ahearn at Martin Marietta to explore the possibility of detecting threading misfit dislocations at Si-GaAs think film interfaces using internal friction methods. If successful, it will be considered as a potential sensor approach for controlling interfacial dislocations during molecular beam epitaxy of thin film compound semiconductors on silicon substrates.

24. NASA/Cal Tech Jet Propulsion Lab (Pasadena, CA)

Dr. Bonnell is collaborating and consulting on design of levitation systems for space applications.

25. National Association of Corrosion Engineers

The NACE-NIST Corrosion Data Center (D.B. Anderson) continues to provide the scientific and technical coordination to the joint program to provide evaluated corrosion data in computerized format. NACE continues to provide a full time Research Associate at NIST plus considerable staff time in support of the program activities. Initial software programs have been well received by industry, with over 600 copies being sold by NACE in the first year. Current activities involve standards activity through ASTM and NACE relating to data formatting, expert system developments for industrial clients, structuring of a comprehensive corrosion database and data entry from two key industrial data collections, development of data evaluation techniques and resolution of technical deficiencies with the corrosion thermodynamics program. Several additional client supported programs are currently under negotiation.

26. New Zealand Department of Scientific and Industrial Research

A cooperative program within the NACE-NIST Corrosion Data Center has been initiated to develop expert systems for materials selection for a variety of critical applications in support of the developing oil and gas industry in New Zealand. The program leader, K.A. Lichti, a

Research Associate from DSIR, is concentrating on materials for subsurface pumps and valves as a function of well defined environmental variables. Basic guidelines are being developed through a review of a wide variety of industrial standards with expert assistance in specific interpretations.

27. Norton Corrosion Limited, Inc., Bothell, WA

E. Escalante supervised and consulted with Norton Corrosion on their application of the computer controlled device, developed in our laboratories, for measuring the corrosion of reinforcing steel in concrete bridge decks.

28. Norris Cylinder, Inc.

A collaborative effort between NIST (J. H. Smith) and Norris Cylinder, Inc. (E. McSweeney) has been initiated to develop light weight high strength steel cylinders and more efficient stainless steel cylinders.

29. Pepco

The Division (G. E. Hicho) has performed numerous failure analyses for the Potomac Electric Power Company (PEPCO) for two reasons: (1) to assist the public utility in its service to the community and (2) to maintain and upgrade NIST competence in materials technology for the electric power industry. As the average age of U.S. plants increases and failures become more frequent, such competence will be needed to identify and transfer state-of-the-art technology to industry.

30. Pratt and Whitney

Haydn Wadley and Robert Schaefer (Processing Group) are collaborating with Pratt and Whitney to determine the HIP mechanism map for a range of intermetallic alloys being considered for high temperature applications in future hypersonic aircraft.

31. Solution Model Database

Groups currently using or evaluating the IMCC model include Dr. Howard Pielet, Inland Steel; Prof. K. S. Spear, Penn State University; Dr. Chad Sheckler, Alfred University; Prof. Paul Daves, Brigham Young University; Prof. Steve Benson, Univ. North Dakota; Prof. Tom Roberts, Milwaukee Area Technical College; Dr. C. David Rogers, USS of USX and Carnegie-Mellon Institute; Prof. Arthur E. Morris, University of Missouri-Rolla.

32. Superconix, Inc.

A collaborative project is underway with C. F. Gallo, Superconix, Inc. and the Magnetic Materials Group (L. J. Swartzendruber) to measure the magnetic properties of superconducting single crystals.

33. Technology Transfer Workshops

R. Ricker presented experimental results obtained at NIST on nickel aluminide corrosion and stress corrosion cracking at a technology transfer workshop at Oak Ridge National Laboratory. The workshops, oganized by ORNL, brought together participants from several companies interested in production, fabrication, and use of nickel aluminide alloys, as well as university and government research scientists.

34. The International Group for Historic Aircraft Recovery (TIGHAR), Mr. R.E. Gillespie, Wilmington, DE

Twelve days before Lindberg's successful crossing of the Atlantic Ocean, two French aviators attempted to fly from France to New York. They never arrived at their destination. It was rumored that they had lost their way and crashed in Maine. TIGHAR believes that they have located the crash site, and they have collected evidence to support this possibility. After 75 years, little of the aircraft is expected to be found, however, TIGHAR found magnetic material at the site that they asked us to examine the material for possible evidence of metals. Using the scanning electron microscope and x-ray fluorescence, the materials was found to contain high concentrations of iron (75.3%) and manganese (5.9%). The material was not metallic, and these concentrations can be found in natural occurring minerals. A more in-depth study is necessary to identify the material. No funding.

35. USX/Carnegie-Mellon

Dr. Rogers of Carnegie-Mellon Institute (formerly U.S. Steel of USX) is developing process models for melt shop use. Dr. Bonnell is working with Dr. Rogers to develop a form of the NIST Steel Slag Model for use as the chemistry component of such process models.

36. Winston-Salem Fire Marshall's Office

Gas from a leaking pipe caused an explosion that demolished a building in North Carolina, and the Corrosion Group was approached to investigate the possible cause of the leak. Examination of the materials brought to us indicated that one section of pipe had undergone extensive corrosion damage. Other sections of pipe failed by mechanical overload from falling debris caused by the explosion. These findings were reported to the Fire Marshall's office for use in their investigation.

37. Zimmer Company, Warsaw, IN

The effects of composition and heat treatment on the mechanical properties of the alloy, Ti-6Al-4V, were studied. The Ti-6Al-4V is used for orthopedic prosthetic devices, and Zimmer is a manufacturer of surgical implants. Transmission microscopy and microstructural analyses of specimens with small compositional changes, heat treated specimens and failed specimens were conducted at NIST.

INDUSTRY/UNIVERSITY

1. BHABHA Atomic Research Center (Government of India)
University of Poona

A cooperative project is underway with the BHABHA Atomic Research Center (Dr. C. K. Gupta) and the University of Poona (Dr. A. P. B. Sinha). This project is part of the Indo-US Physical, Materials and Marine Sciences Collaboration Program and the objective of this project is to study the influence of nitrogen content on the stress corrosion cracking behavior of stainless steels (alloy 316L). NIST has provided material for this study and complimentary experiments will be conducted at the various institutions. A workshop on the Corrosion Science and Technology on Stainless Steels is being planned to be held in India.

2. Lehigh University/Intergraph Corp.

R. J. Fields continued his collaboration with Dr. T. -S. Liu of Intergraph and Prof. T. J. Delph of Lehigh on the quantification of creep damage in metals. Results of recent work were published in Acta Metallurgica. R. J. Fields is now measuring cavities in-situ using synchrotron radiography. The bicrystals for this work are creep stressed at Lehigh University.

3. NIST Metals Processing Laboratory

The Metals Processing Laboratory of the National Institute of Standards and Technology contains facilities for preparation of special samples not readily obtainable elsewhere. Scientists from industry and universities can come to NIST to help prepare samples for independent or collaborative research. During the past year, investigators from Crucible Materials, General Electric, National Aeronautics and Space Administration, Johns Hopkins University, University of California, and University of Wisconsin have participated in interactions in this program.

4. SRI (Stanford Research International)

Drs. Hastie and Bonnell are collaborating with Dr. D. Hildenbrand of SRI in the mass spectral analysis of complex high temperature vapors. NIST and SRI are using techniques which are unique to each laboratory but are nevertheless complementary.

UNIVERSITIES

1. Applied Physics Laboratory, The Johns Hopkins University

A collaborative effort is underway between the Applied Physics Laboratory of the Johns Hopkins University (K. Moorjani) and NIST (R. Shull) to prepare and investigate the magnetic behavior of composite materials having nanocrystalline-sized grains.

2. Applied Physics Laboratory, The Johns Hopkins University

   A collaborative effort is underway between the Applied Physics Laboratory of the Johns Hopkins University (J. Bohandy) and NIST (L. Bennett) to prepare high T_c superconductors by laser ablation and investigate their magnetic properties.

3. Ben-Gurion University

   A Joint U.S. Israeli Binational Science Foundation program on solid state amorphization transformations is underway between NIST (D. S. Lashmore) and Ben-Gurion University (M. P. Dariel).

4. Boris Kidric Institute (BKI), Belgrade, Yugoslavia

   A collaborative activity is underway between Dr. Hastie and Dr. Zmbov of BKI for the mass spectrometric analysis of complex high temperature processes, including plasma deposition of amorphous silicon.

5. Cambridge University

   Professor Michael F. Ashby and graduate students at Cambridge University (England) are developing predictive models for the hot isostatic pressing of intermetallic composites in collaboration with Metallurgy Division scientists.

6. Carnegie Mellon University

   NIST and ~~General Electric~~ researchers are collaborating with Professors M. Fox and I. Hultage in CMU's Robotics Institute to explore the application of artificial intelligence techniques in design/redesign of materials processing pathways.

7. Free University of Brussells

   Drs. Hastie and Bonnell are collaborating with Dr. J. Drowart (Brussels) on a survey of ionization cross section usage in high temperature mass spectrometry.

8. Georgia Institute of Technology

   Professor Shui-Nee Chow and coworkers are developing new mathematical techniques for studying dynamical systems and applying them to the spinodal decomposition of alloys.

9. George Mason University

   Professor Raymond Chapman and coworkers are exploring new methods of conflict resolution and applying them to the development of multi-group research programs such as DARPA's program on Innovative Concept for Design (DICE).

10. George Washington University

George Hicho serves as an Associate Professorial Lecturer in Engineering at George Washington University in the area of Mechanical Properties testing.

11. Indira Gandhi Institute (IGI) Kalpakkam, India

A collaborative activity is underway between Dr. Hastie and Dr. Mathews of IGI for the mass spectrometric investigation of materials at very high temperatures generated by laser heating.

12. Institute of Metals Research, Academia Sinica, Shenyang

A collaborative effort is underway with a guest scientist, Prof. Y. Huang, of the Institute of Metals Research, Academia Sinica, Shenyang, Peoples Republic of China, to investigate the fracture of metals using infrared thermography and DC potential drop techniques to measure crack length.

13. Johns Hopkins University

Studies on the mechanism of transgranular stress corrosion cracking have been pursued in cooperation with Johns Hopkins University (Dr. J. Kruger). The experimental studies were carried out at NBS by a graduate student (T. Cassagne), and the staff of the Corrosion Group was involved both in the experimental and in the analysis of the results.

14. Johns Hopkins University

A joint investigation of the mechanical properties of electrodeposited CMS alloys is being conducted with Johns Hopkins University in cooperation with Dr. Moshe Rosen. (D. S. Lashmore and R. R. Oberle)

15. Johns Hopkins University

A collaborative effort with Johns Hopkins University (D. Shectman) which resulted in the discovery of an entirely new class of materials, the quasicrystals, was continued. Emphasis has been on studies of the structure of these unusual alloys and the processing conditions that produce them.

16. Korean Advanced Institute of Standards and Technology

A collaborative effort is underway between Alexander Shapiro of the Metallurgy Division and a guest scientist, Prof. Duk H. Yoou, of the Korean Advanced Institute of Standards and Technology (KAIST) to study grain boundary migration in (6 MgO + 4 CuO) + 90 ZrO_2.

17. Northwestern University

Studies of coarsening rates in Pb-Sn solid-liquid mixtures are being performed cooperatively with Northwestern University (P. Voorhees). The aim of this work is to test and develop general rules that will be applicable to a large variety of metallurgical processes.

18. Oregon Graduate Research Center

A collaborative effort has been initiated between NIST (J. H. Smith) and the Oregon Graduate Research Center (Prof. Atteridge) to develop test methods and standards for fracture resistant high strength steel cylinders.

19. Rice University

Dr. Bonnell continues to provide consultation support to the High Temperature Group in the Chemistry Department on levitation and thermophysical properties of liquid metals.

20. University of Maryland

R. deWit and R. J. Fields collaborated with G. R. Irwin and X. J. Zhang of the University of Maryland in the understanding of cleavage crack arrest and reinitiation in steel. Quantitative topological of fracture surfaces from wide-plate crack-arrest test were made at NIST and analyzed at the University of Maryland. These topographies provided corroboration for the very high crack arrest toughness found in the tests. Continued interaction with University of Maryland seeks to correlate reduction-in-thickness contours measured at NIST with the topographic results.

21. University of Pennsylvania

R. J. Fields collaborated with Prof. David Pope and John Benci of the University of Pennsylvania in the quantification of creep damage in non-uniform, multiaxial stress fields. Bars of a heat-resisting, ferritic alloy containing notches of varying acuity were creep damaged to various degrees. Synchrotron x-radiographs were made in the region of the notches. Quantitative image analysis of these radiographs permitted contours of constant creep damage to be determined around the notches. Such quantitative damage maps are making possible the critical assessment of various creep damage theories.

22. University of Science and Technology - Beijing, China

In a program funded by the United Nations Industrial Development Organization (UNIDO), a seminar series on corrosion database development was provided by D. B. Anderson at UST-Beijing to an audience of students and corrosion researchers from throughout China. The program has also

funded a fellowship for a Guest Researcher (Dr. Changrong Li) for a one year visit in the Corrosion Data Center where her goal is to develop a prototype database addressing key Chinese industrial development needs.

23. University of Wisconsin at Madison

Studies of microstructure development and phase diagram relationships in intermetallics have been expanded and applied to new materials as a result of cooperative programs underway with J. Perepezko and A. Chang of the University of Wisconsin.

TECHNICAL/PROFESSIONAL COMMITTEE LEADERSHIP ACTIVITIES

Alpha Sigma Mu, The Metallurgy and Materials Honor Society
R. E. Ricker, National Board of Trustees

American Association for Crystal Growth
S. R. Coriell, Executive Committee

American Electroplaters and Surface Finishers Society
C. E. Johnson, Electrocomposites Committee
D. S. Lashmore, Alloy Deposition Subcommittee

American Institute of Mining, Metallurgical and Petroleum Engineers
The Metallurgical Society
J. B. Clark, The Physical Metallurgy Committee
J. B. Clark, The Physics and Chemistry of Materials Committee
R. E. Ricker, Committee on Corrosion and Environmental Effects
H.N.G. Wadley, Secretary, Materials Design and Manufacturing Division

American Petroleum Institute
Task Group on Tankage Brittle Fracture
J. H. Smith

Applied Mechanics Reviews
H.N.G. Wadley, Associate Editor

ASM INTERNATIONAL
Alloy Phase Diagram Appropriations Committee
E. N. Pugh

Alloy Phase Diagram Data Committee
J. B. Clark, Chairman

Corrosion and Environmental Effects Committee
R. E. Ricker

Editorial Committee for Advanced Materials and Processes
E. N. Pugh

Materials Science Division Council
J. B. Clark

NDT Committee
H.N.G. Wadley

Subcommittee on Metallurgical Reactions and Electromigration in Electronic Devices
C. A. Handwerker, Chairman

Technology Transfer Committee
E. N. Pugh

Washington, DC Chapter - Education Committee
R. E. Ricker, Chairman

ASTM

Committee on Terminology
C. G. Interrante, Technical Committee Representative

Coordinating Group on Terminology for Environmental Activities
C. G. Interrante, Convener

B5:	Copper and Copper Alloys L. J. Swartzendruber
B7:	Light Metals R. D. Shull W. J. Boettinger
B8: B8.10:	Metallic and Inorganic Coatings General Test Methods C. E. Johnson D. S. Lashmore
B8.10.03:	Microhardness Testing C. E. Johnson, Liaison to E04 D. S. Lashmore, Liaison to E04
C26: C26.07:	Nuclear Fuel Cycle Waste Materials C. G. Interrante
C26.13:	Repository Waste Package Materials Testing C. G. Interrante, Leader, Task Group on Terminology
E3: E3.07:	Chemical Analysis of Metals Acoustic Emission R. C. Clough
E4: E4.05:	Metallography Microhardness C. E. Johnson D. S. Lashmore
E7:	Nondestructive Testing L. J. Swartzendruber L. H. Bennett
E7:04	Acoustic Emission J. A. Simmons R. C. Clough

E24:	Fracture Testing C. G. Interrante, Member of Executive Committee
E24:02	Fractography and Associated Microstructures G. E. Hicho
E24.04:	Sub-Critical Crack Growth C. G. Interrante
E24.05:	Terminology for Fracture Testing C. G. Interrante, Co-Chairman R. deWit, Secretary
E24.06:	Fracture Mechanics Applications J. H. Smith G. E. Hicho
E28: E28.06.07:	Mechanical Testing Hardness Test Block Intercomparison Task Group T. R. Shives, Chairman
E49:	Computerization of Material Property Data D. B. Anderson, Steering Committee for Second International Symposium on Computerized Materials Databases
E49.02.5:	Corrosion Data Formats D. B. Anderson, Chairman
E49:91:	D. B. Anderson, Awards Committee Chairman
F4: F4.02.09:	Medical and Surgical Materials and Devices Joint Section on Corrosion of Implants A. C. Fraker, Co-Chairperson
F7: F7.04:	Aerospace Industry Methods Hydrogen Embrittlement Testing C. G. Interrante, Chairman, Task Group on Terminology
G1: G1.03.1:	Corrosion of Metals Corrosion Data Formats Task Group D. B. Anderson, Chairman
G1.06:	SCC and Corrosion Fatigue R. E. Ricker C. G. Interrante
G1.10:	Corrosion of Metals in Soil E. Escalante
G1.10.01:	Measurement of pH of Soil E. Escalante, Task Group Leader

G1.10.02: Measurement of Soil Resistivity
E. Escalante

G1.11: Electrochemical Measurements in Corrosion
R. E. Ricker

G1.14: Corrosion of Steel in Concrete
E. Escalante

G1.99: D. B. Anderson, Liason to ASTM Committee E-49

Electrochemical Society
Deposition from Non-Aqueous Electrolytes Symposium
G. R. Stafford, Co-Chairman

Electrodeposition Division
D. S. Lashmore, Chairman, Editor

Hydrogen Production and Storage Symposium Co-Chairman
D. E. Hall

Industrial Electroytic Division
D. E. Hall, Vice Chairman and Executive Committee

Symposia Subcommittee
D. S. Lashmore

Federation of Materials Societies
S. R. Coriell, Trustee

High Temperature Science Journal
J. W. Hastie, Editorial Board Member

International Advisory Committee and Program Committee for International Conference on Rapidly Quenched Metals
W. J. Boettinger, Member

International Committee for Conference on Hot Isostatic Pressing
R. J. Schaefer, Chairman

International Standards Organization
TC107: Metallic and Other Non-Organic Coatings
C. E. Johnson, Delegate

TC107.02: Methods of Inspection and Co-Ordination of Test Methods
C. E. Johnson, Delegate

TC107.03: Electrodeposited Coatings and Related Finishes
C. E. Johnson, Delegate

TC164: Mechanical Testing
1. Terminology
C. G. Interrante, Delegate

International Union of Pure and Applied Chemistry Commission II-3
High Temperature and Solid State Chemistry
J. W. Hasties, Titular Member

Maryland Institute of Metals
W. J. Boettinger, Executive Board Member

Materials Science and Engineering
L. H. Bennett, Associate Editor

Mechanical Failures Prevention Group
T. R. Shives, Executive Secretary and Editor of Proceedings

National Aeronautics and Space Administration
High Temperature Advisory Committee
D. W. Bonnell, Member

National Association of Corrosion Engineers (NACE)
T.3 Corrosion Science and Technology Committee
T.3E: Unit Committee on SCC and Corrosion Fatigue
R. Ricker

T.3K: Corrosion and Other Deterioration Phenomenon Associated with Concrete
E. Escalante

T.3L: Unit Committee on Electrochemical Techniques
R. Ricker

T.3U.1: Task Group on Corrosion Data Formats
D. B. Anderson, Chairman

NACE Board of Directors
E. N. Pugh, Ex Officio Member

NACE Research Committee
E. N. Pugh, Chairman

NACE-NIST Corrosion Data Program
Administrative Committee
D. E. Hall
D. B. Anderson

Board of Trustees
E. N. Pugh

NACE Baltimore-Washington Chapter Executive Committee
R. Ricker, Membership Committee Chairman

National Research Council
Transportation Research Board
E. Escalante, Panel Member

Office of Energy-Related Inventions, National Bureau of Standards
L. H. Bennett, Reviewer of Invention Disclosures
J. H. Smith, Reviewer of Invention Disclosures

Society for Biomaterials Standards
A. C. Fraker, Standards Representative on the ASTM F4 Committee

Society of Automotive Engineers/ASTM
Unified Numbering System for Metals and Alloys
L. H. Bennett, NBS Representative

U.S. Department of Transportation
Technical Hazardous Liquid Pipeline Safety Standards Committee
R. J. Fields, Secretary

1989 International Conference on High Temperature Materials Chemistry
J. W. Hastie, Chairman
D. W. Bonnell, Vice Chairman

U.S. DEPARTMENT OF COMMERCE
National Institute of Standards and Technology

DIRECTOR
Deputy Director
- Office of Congressional and Legislative Affairs
- Associate Director for International and Academic Affairs
- Associate Director for Industry and Standards

ASSOCIATE DIRECTOR FOR PROGRAMS, BUDGET, & FINANCE
- Program Office
- Budget Office
- Office of the Comptroller

OFFICE OF THE DIRECTOR NBS/BOULDER LABORATORIES

DIRECTOR OF ADMINISTRATION
- EEO Support
- Information Resources & Services
- Public Information
- Management Systems
- Plant
- Facilities Services
- Occupational Health & Safety
- Personnel
- Acquisition and Assistance

NATIONAL MEASUREMENT LABORATORY
- Office of Standard Reference Data
- Office of Measurement Services
- Center for Basic Standards
- Center for Radiation Research
- Center for Chemical Physics
- Center for Analytical Chemistry

NATIONAL ENGINEERING LABORATORY
- Center for Applied Mathematics
- Center for Electronics & Electrical Engineering
- Center for Manufacturing Engineering
- Center for Building Technology
- Center for Fire Research
- Center for Chemical Engineering

NATIONAL COMPUTER AND TELECOMMUNICATIONS LABORATORY
- Information Systems Engineering Division
- Systems and Software Technology Division
- Computer Security Division
- System and Network Architecture Division
- Advance Systems Division

INSTITUTE FOR MATERIALS SCIENCE & ENGINEERING
- Office of Nondestructive Evaluation
- Ceramics Division
- Fracture and Deformation Division
- Polymers Division
- Metallurgy Division
- Reactor Radiation Division

Institute for Materials Science and Engineering

L. H. Schwartz, Director
H. L. Rook, Deputy Director

Nondestructive Evaluation
H. T. Yolken, Chief
L. Mordfin, Deputy

Institute Scientists
J. W. Cahn
R. M. Thomson
S. M. Wiederhorn

Metallurgy
E. N. Pugh, Chief
J. H. Smith, Deputy

Polymers
L. E. Smith, Chief
B. M. Fanconi, Deputy

Ceramics
S. M. Hsu, Chief
S. J. Dapkunas, Deputy

Fracture and Deformation
H. I. McHenry, Chief
Deputy, Vacant

Reactor Radiation
R. S. Carter, Chief
T. M. Raby, Deputy

NBS-114A (REV. 2-80)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET (See instructions)	1. PUBLICATION OR REPORT NO. NISTIR 88-3843	2. Performing Organ. Report No.	3. Publication Date December 1988

4. TITLE AND SUBTITLE

Institute for Materials Science and Engineering; Metallurgy Division, Technical Activities 1988

5. AUTHOR(S)

6. PERFORMING ORGANIZATION (If joint or other than NBS, see instructions)

NATIONAL BUREAU OF STANDARDS
DEPARTMENT OF COMMERCE
WASHINGTON, D.C. 20234

7. Contract/Grant No.

8. Type of Report & Period Covered

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP)

10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

This report summarizes the FY 1988 activities of the Metallurgy Division of the National Institute of Standards and Technology (NIST). The research centers upon the structure-processing-properties relations of metals and alloys and on the methods of their measurement. The activities also include the generation and evaluation of critical materials data. Efforts comprise studies of metallurgical processing, corrosion, mechanical properties, electrodeposition, process sensors, high temperature reactions and magnetic materials.

The work described also includes four cooperative programs with American professional socities and industry: the National Association of Corrosion Engineers (NACE) - NIST Corrosion Data Program, the Aluminum Association - NIST Temperature Sensor Program, the American Iron and Steel Institute (AISI) - NIST Steel Sensor Program, and the ASM INTERNATIONAL (ASM) - NIST Alloy Phase Diagram Program.

The scientific publications, committee participation, and other professional interactions of the 79 full-time and part-time permanent members of the Metallurgy Division and its 48 guest researchers are identified.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key-words by semicolons)

Corrosion; Electrodeposition; Magnetic Properties; Metals Processing; Metallurgy; Process Sensors

13. AVAILABILITY

☒ Unlimited
☐ For Official Distribution. Do Not Release to NTIS
☐ Order From Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.
☒ Order From National Technical Information Service (NTIS), Springfield, VA. 22161

14. NO. OF PRINTED PAGES

125

15. Price

$18.95